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International Comparison of Guarded Hot Plate Apparatus Using National and Regional Reference Materials

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LNE LABORATOIRE NATIONAL D'ESSAIS

NPL National Physical Laboratory

National Research Council Canada / Conseil national de recherches Canada

Institute for Reference Materials and Measurements

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[1] At Boulder, CO 80303.
[2] Some elements at Boulder, CO.

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ABSTRACT

As part of an international comparison of guarded-hot-plate apparatus, thermal conductivity measurements of four different thermal insulation materials are presented. The test program evaluates one regional and three national reference materials using a two-part experimental plan: 1) five independent replicate measurements taken at a fixed temperature (297.15 K); and, single-point measurements taken at multiple temperatures (280 K to 320 K). The analysis of the replicate data provides rankings for the primary factors – laboratory and material. A major finding of the replicate analysis is the existence of a laboratory-material interaction. In other words, there are laboratory-to-laboratory changes in both location and variation, which change from material to material. Further analyses attempt to determine the sources (i.e., underlying causes) for the variation in the replicate data. Secondary laboratory factors are investigated both individually following a cause-and-effect chart, and collectively using graphical analysis, correlation analysis and analysis of variance. The major finding of the multi-temperature (280 K to 320 K) analysis confirms and supports the laboratory-material interaction as found in the fixed-temperature replicate data analysis. The multi-temperature analysis also reveals an increasing difference between laboratories as the mean specimen temperature departs from 297.15 K.

KEY WORDS: comparison, density, certified reference material, expanded polystyrene, fibrous glass, exploratory data analysis, graphical analysis, guarded hot plate, interlaboratory, repeatability, SRM, thermal conductivity, thermal insulation

Table of Contents

Table of Contents (continued)

List of Tables

List of Figures

1 Introduction

1.1 Problem Definition

A recent ASTM C-16 Workshop [1] on thermal insulation standard reference materials identified the transferal of national reference materials and their acceptance across international boundaries as a potential obstacle to global commerce. This (apparent) limitation with national and regional reference materials and the continued expansion of global commerce has motivated several national standards laboratories to develop a technical exchange of information by conducting international comparisons. The purpose of this international comparison is to assess the measurement variability among test results of different guarded-hot-plate apparatus located in national standards laboratories in Canada, France, Japan, United Kingdom, and United States. The laboratory participants were the National Research Council Canada (NRCC), Laboratoire National d'Essais (LNE), Japan Testing Center for Construction Materials (JTCCM), National Physical Laboratory (NPL), and National Institute of Standards and Technology (NIST), respectively. The European Commission Institute for Reference Materials and Measurements (IRMM) provided one of the reference materials and was an official observer.

1.2 Comparison Process

In order to foster confidence and credibility in these international measurements, this comparison was conducted using four different national reference materials. Two of the reference materials were issued by NIST; one by the European Commission IRMM; and one by JTCCM. Ten specimens from each lot of material were selected and characterized by the issuing organization (or delegate laboratory), paired by density, and a single specimen pair distributed to each participant. The laboratory participants were requested to conduct five replicate measurements of each material at a fixed temperature of 297.15 K (24 °C). In addition, each recipient laboratory measured the each material at five specified temperatures from 280 K to 320 K. The tests were to be conducted using either Test Method ISO 8302 [2] or ASTM C 177 [3]. All test data were recorded on official test forms and reported to NIST for analysis. This study was organized in 1997 by NPL; material characterization by the issuing laboratories was completed in 1998; participant measurements completed in 1999; and the resulting data analyzed in 2000 by NIST.

1.3 Background

An interlaboratory, or collaborative, test program provides the participants with the means of 1) assessing the clarity of a particular test method, 2) verifying that the results are in agreement with accuracy statements specified by the method, and 3) maintaining a periodic check of a group of laboratories [4,5]. These objectives have motivated several recent international comparisons of guarded hot plate apparatus [6-8]. Each comparison has investigated a relatively small number of thermal insulation materials over a specified temperature range (typically at or near 297 K). The comparisons have endeavored to minimize issues of material variability by circulating the same pairs of thermal insulation materials among the laboratories following the strict format of a "round robin" test program. Replicate measurements at fixed levels of temperature were not requested. Although desirable, increasing the number of materials and replicates adds a considerable burden to the participating laboratory and increasing the number of laboratories adds a considerable burden to the coordination of the collaboration.

One of the most ambitious studies [6] was organized in 1978 and involved nearly 50 guarded-hot-plate laboratories from Africa, Asia, Australia, Europe and North America. The study was intended to determine the worldwide state-of-the-art in guarded hot plate measurements prior to the development of ISO standards. Because of the large number of laboratories involved, this study can be considered the starting point for all modern international comparisons of guarded-hot-plate apparatus. Participants measured the thermal conductivity of fibrous glass specimens at mean temperatures of 283 K, 297 K, and a third temperature within the range from 273 K to 313 K. The results indicate that the relative standard deviation of the data from the fitted curve is 2.4 % [6], although several data points deviate from the curve by more than 5 % and some by more than 10 %. The original test plan envisioned a second comparison similar in scope to the initial study to determine if an improvement in precision was realized after the approval of ISO 8302 [2]. To date, the follow-up study has not been completed.

More recent international comparisons [7,8] of guarded-hot-plate apparatus have emphasized a smaller number of laboratories, generally with participation from at least two national standard laboratories. In one study [7], three pairs of glass-fiber specimens having thicknesses of 26 mm and 75 mm and bulk densities of 44 kg/m^3 and 53 kg/m^3 were circulated to three laboratories. In the worst case, the relative standard deviation of the data from the fitted curve fit is less than 1.5 % and the largest difference between the sets of measurements is 2.2 % [7]. In the second study [8], one pair of glass-fiber specimens (159 kg/m^3) and fibrous alumina silica (288 kg/m^3) was circulated between two national standard laboratories in North America. The standard deviations, multiplied by 2, for the fitted models of glass-fiber board and fibrous alumina silica board are 1.4 % and 0.5 % [8], respectively. Both of these studies concluded that, although some small systematic differences were present among the laboratories, the variations of the test data were less than the imprecision levels of ± 2 % to ± 5 % currently specified in ISO 8302 [2] and ASTM C 177 [3].

1.4 Overview

This comparison is intended as another step in assessing the variability among international guarded-hot-plate laboratories. In contrast to the "round-robin" format of the earlier studies [6-8], this comparison distributed individual specimens of four different national reference materials to each participant for measurement. Here, the primary goal is to assess and quantify the level of variability in the guarded-hot-plate measurements and to encourage confidence and credibility in international measurements of different national reference materials. This report describes the test program, guarded hot plate laboratories, reference materials and their certification equations, test method and equipment, material characterization, test results, and data analysis. The data analyses include both statistical and engineering assessments. Final rankings for the materials and laboratories are summarized, and recommendations for future comparisons are given.

2 Test Program

This section describes the specific objectives of the test program in context with the overall goal of the comparison. Technical contacts and addresses are provided for the laboratories as well as a technical description of the reference materials and certified equations (where appropriate). A summary of the test protocol given to the participants is also provided.

2.1 Purpose and Objectives

The purpose of this interlaboratory comparison is to investigate the variability in test results among guarded hot plate laboratories in Canada, France, Japan, United Kingdom, and United States using different national reference materials. There are three primary objectives:

1) Characterize the material properties, specifically the bulk density and thermal conductivity, of each sample of specimens distributed to the laboratories;
2) Quantify and assess the level of variability (both within- and between-laboratory) of the fixed temperature (297.15 K) replicate data; and
3) Quantify the effect of temperature by comparison of the multi-temperature (280 K to 320 K) data.

2.2 Laboratory Participants

Table 1 summarizes the participant laboratory's organization, country, and contact(s). The addresses of the participants are provided in Appendix A. The final list of participants resulted from a series of informal communications, meetings, and subsequent proposals initiated by the National Physical Laboratory (NPL). In 1997, the National Institute of Standards and Technology (NIST) agreed to assist in planning the interlaboratory comparison and to provide statistical analyses of the test data.

TABLE 1 – Laboratory Participants

Organization	Country	Contact
1) Japan Testing Center for Construction Materials (JTCCM)	Japan	Masayoshi Uezono
2) Laboratoire National d'Essais (LNE)	France	Gianni Venuti, Sylvie Quin
3) National Institute of Standards and Technology (NIST)	United States	Robert Zarr
4) National Physical Laboratory (NPL)	United Kingdom	David Salmon, Ronald Tye
5) National Research Council Canada (NRCC)	Canada	Kumar Kumaran, Fitsum Tariku
European Commission Institute for Reference Materials and Measurements (IRMM) (Observer only)	European Commission	Jean Pauwels

2.3 Reference Materials

The reference materials were selected to obtain a wide range – yet manageable number – of insulation materials from Asia, Europe, and North America. Table 2 summarizes the reference materials by designation, material description, (nominal) density, (nominal) thickness, temperature range, and source. Materials 1, 2, and 3 were fibrous insulations covering a broad range of densities; material 4 was a molded beads, expanded polystyrene board. Material 3, which is a mixture of glass and mineral oxides fibers having high temperature capabilities, is currently undergoing an internal review process for certification. Each source laboratory was responsible for characterizing 10 specimens of the reference material. In 1998, the European Commission IRMM agreed to provide specimens of IRMM-440 (replacement lot for Certified Reference Material 064) to NPL for characterization and distribution to the participants. As a side note, the NIST Standard Reference Material Program (SRMP) has officially designated SRM 1451 as obsolete due to historically low customer demand. NIST SRMP continues to offer SRM 1452, which is from the same material lot as 1451. Specimens of SRM 1451 are currently available from the NIST Building and Fire Research Laboratory.

TABLE 2 – Reference Materials

Designation	Description	Density (kg/m³)	Thickness (mm)	Temperature (K)	Source and Ref.
1) SRM† 1451	Fibrous glass blanket	13	25	100 to 330	NIST [9]
2) IRMM-440‡	Resin-bonded glass fibre board	70	35	263 to 323	IRMM (via LNE [10])
3) JTCCM candidate	Mineral-oxide fiber board	200	25		JTCCM
4) SRM 1453	Expanded polystyrene board	38	13	285 to 310	NIST [11]

†Standard Reference Material (SRM) issued by NIST.

‡Certified Reference Material (CRM) issued by IRMM.

2.4 Certified Values for Reference Materials

A Certified Reference Material (CRM) is accompanied by a certificate having one or more property values certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence [12]. Certified values of thermal conductivity for SRM 1451, IRMM-440, and SRM 1453 are described by the following general model modified from Reference [9]:

$$\lambda(T_m, \rho) = a_0 + a_1\rho + a_2 T_m + a_3 T_m^2 + a_4 \frac{T_m^3}{\rho} + a_5 e^{-\left[\frac{(T_m - 180)}{75}\right]^2} \quad (1)$$

Here, $\lambda(T_m, \rho)$ is predicted thermal conductivity (W/m·K); ρ is the bulk density (kg/m³); and, T_m is the mean specimen temperature (K). The thermal transmission properties of heat insulators determined from standard test methods typically include several mechanisms of heat transfer, including conduction, radiation, and possibly convection. For that reason, some experimentalists will include the adjective "apparent" when describing thermal conductivity of thermal insulation. However, for brevity, the term thermal conductivity will be used in this report.

Table 3 summarizes the regression coefficients (a_i) and expanded uncertainties (U) at a coverage factor of k = 2 for predicted values of IRMM-440, and SRM 1453. For SRM 1451 [9], the uncertainty statement of ± 3 % was developed before current international guidelines on expressing uncertainty became effective; therefore, a coverage factor is unavailable.

TABLE 3 – Regression Coefficients (Eq 1) for SRM 1451, IRMM-440, and SRM 1453

Designation	a_0	a_1	a_2	a_3	a_4	a_5	U (k=2)
SRM 1451	-1.059×10^{-4}	1.378×10^{-4}	7.714×10^{-5}	0	8.472×10^{-9}	1.339×10^{-3}	± 3 %†
IRMM-440‡	0.0293949	0	0.0001060	2.047×10^{-7}	0	0	± 1 %
SRM 1453	6.3054×10^{-4}	-4.1993×10^{-5}	1.1650×10^{-4}	0	0	0	± 1.3 %

†Coverage factor is unavailable.

‡Temperature units are in [°C], not [K].

The official certification of IRMM-440 was finished after the completion of the data analysis of this interlaboratory comparison. Thus, the certified equation and regression coefficients for IRMM-440 are presented only for comparison purposes. The JTCCM "candidate" reference material is currently undergoing certification processes and, consequently, certified values of thermal conductivity are unavailable.

2.5 Protocol

Using test methods ISO 8302 [2] and ASTM C 177 [3], a short test protocol was drafted and subsequently approved by the participants for this comparison (see Appendix B). The protocol was partitioned into two sections. The first section covered the characterization by the source laboratories providing reference materials and the second section covered the measurements conducted by the participants. Each source laboratory provided one pair of specimens of reference materials to each participant (5 pairs in total). In order to assess the material variability, the density and thermal conductivity of each specimen pair was measured by the source laboratory at a mean temperature of 297.15 K (24 °C) and temperature difference of 20 K (n = 5 measurements per material). Needless to say, the logistics of providing reference measurements and subsequently delivering all specimens to the participants was time consuming.

The test protocol for the participants required a series of replicate and multi-temperature measurements for each material. Initially, the thermal conductivity of each pair of specimens was determined five times at a (fixed) mean temperature of 297.15 K (24 °C) and a temperature difference of 20 K (n = 20 measurements for 4 materials). The operator was required to remove the specimens from the apparatus after each measurement and re-install the specimens after sufficient conditioning. After completion of the replicate measurements, thermal conductivity measurements were conducted for each material at 280 K, 290 K, 300 K, 310 K, and 320 K and a temperature difference of 20 K (n = 20 measurements for 4 materials). Note that although the multi-temperature tests were conducted in random order, the specimens were not removed from the apparatus between temperature settings.

With exception of SRM 1451, the materials were to be tested at thicknesses determined by each laboratory with the only provision that the pressure exerted on the specimens by the measuring equipment was limited to a range between 1000 Pa and 2000 Pa. For SRM 1451, the test thickness was limited to 25.4 mm by utilizing spacer stops placed at the perimeter of the specimen to prevent over compression of the material during testing. The use of spacer stops for the other materials (for example, limiting plate movement due to specimen creep, if any) was left to the operator's discretion. The material characterization and test data were recorded in SI units on "official" data entry forms and returned to NIST. An annotated copy of each form is given in Appendices C and D, respectively. Upon receipt by NIST, the test data were input into electronic spreadsheets, checked for consistency, and subsequently analyzed using graphical exploration techniques.

3 Test Method and Apparatus

3.1 Operating Principle

Measurements of thermal conductivity were determined in accordance with procedures in standard test methods, ISO 8302 [2] and/or ASTM C 177 [3], which are summarized briefly here. For the double-sided mode of operation, two specimens having the same density, size, and thickness are placed on the two sides of the guarded hot plate and clamped securely by the cold plates. Ideally, the guarded hot plate and the cold plates provide constant temperature boundary conditions to the surfaces of the specimens. With proper guarding in the lateral direction, the

apparatus is designed to provide one-dimensional heat flow (Q) through the meter area of the pair of specimens. Additional guarding is provided by means of a temperature controlled environmental chamber, edge insulation, linear guarding, or a combination of these. When in use, the environmental chamber ordinarily maintains the ambient air temperature at the same value as the mean temperature (T_m) of the hot and the cold plates.

Under steady-state conditions, measurements of thermal conductivity (λ) for the pair of specimens are determined using the following equation:

$$Q = \lambda \, 2A \frac{\Delta T}{L} \tag{2}$$

where Q is the heat flow through the meter area of the specimens (W); $2A$ is the meter area normal to direction of heat flow, both sides (m^2); ΔT is the temperature difference across the hot (T_h) and cold surfaces (T_c) of the specimens (K); and, L is the in-situ thickness of the pair of specimens (m). Values of λ are typically reported at the mean temperature of the hot and cold plates, $T_m = \frac{1}{2}(T_h + T_c)$.

For a single-sided mode of operation, a single specimen (selected from the pair) is placed between the hot and cold plates of the apparatus. The other specimen is replaced with an auxiliary piece of insulation that is placed between the hot and guard plate. The auxiliary guard plate is maintained at the same temperature as the hot plate. With proper guarding in the lateral direction, the apparatus is designed to provide one-dimensional heat flow (Q) through the meter area of the single specimen. For determining λ in the single-sided case, Eq 2 is modified slightly by taking a meter area (A) coefficient of unity. In general, all other particulars of the test procedure are essentially the same as the double-sided mode of operation.

3.2 Equipment

Table 4 summarizes the pertinent features of the guarded hot plate (GHP) apparatus used in this comparison as reported by the laboratory participants. The information covers four categories: 1) apparatus manufacturer, 2) principle (i.e., test method utilized), 3) relative expanded uncertainty for the estimate of λ, and 4) details of the apparatus components including plate sizes, emittance, heaters, guarding, and temperature sensors. Most laboratories fabricated their own apparatus; however, in one case (JTCCM), a commercial vendor fabricated one of the apparatus. The laboratories conducted their tests utilizing the guarded hot plate principle, following either the ISO 8302 [2] or ASTM C 177 [3] test method.

3.2.1 Uncertainty: The relative expanded uncertainty [13] (k =2) associated with measurement estimates of λ for each apparatus range from 1.0 % to 1.5 % (Table 4) and is used later for the engineering analysis of the test data. One laboratory (JTCCM) did not report their estimated uncertainty. The expanded uncertainty (U) defines an interval about the result of a measurement (y) that may be expected to encompass a large fraction of the distribution of values ($Y = y + U$) that could reasonably be attributed to the measurand (in this case, λ). Here, the expanded uncertainty corresponds to a level of confidence of 95 % by taking a coverage factor of $k = 2$ [13]. The relative expanded uncertainty is defined as $U/|y|$, $|y| \neq 0$ [13].

TABLE 4 – Guarded Hot Plate Apparatus

	JTCCM	LNE	NIST	NPL	NRCC
Manufacturer	Eko Instruments Trading Co., Ltd.	LNE	NIST	NPL	NRCC
Principle	GHP	ISO 8302	ASTM C 177	ISO 8302	ASTM C 177
Relative Expanded Uncertainty (k=2) (%)	Not reported	1.5	1.5 (IRMM-440) 1.0 (other)	1.2	1.0
Plate size (mm)	300×300	610×610	1016 diameter	610×610	610×610
Meter plate size (mm)	150×150	300×300	406.4 diameter	305.2×305.2	250×250
Plate material	Al	Al	Al	Hot – Cu Cold – Al	Al
Plate emittance	0.9	0.86 ± 0.05 (k=2)	0.89	>0.9	0.89
Type of heater	Distributed	Distributed[1]	Line source[2]	Distributed[3]	Distributed
Edge guarding	Condition air	Note[4]	Condition air	Note[5]	Glass-fiber
Temperature sensor	Type T	Type K	PRT	Type E	Type T
Operation mode	2-sided	2-sided	2-sided	1-sided	2-sided

[1]Isolated Ni/Cr heating resistance with a central metering area and a guard section
[2]Line-heat source per ASTM C 1043 [14]: 1 ribbon heater in the meter plate, 2 swaged heaters in the guard ring
[3]Double sided copper printed circuit board
[4]Edge insulation, temperature controlled peripheral guard and additional outer edge insulation
[5]Linear temperature gradient edge guard and 100 mm expanded polystyrene

3.2.2 Components: As noted in Table 4, there is a wide variety in the design of the apparatus components. The plates range in size (and geometry) from 300 mm square to 1016 mm in diameter. Three of the apparatus plates, however, are 610 mm square. All of the apparatus have different meter plate sizes (albeit the differences for LNE and NPL are small). Most of the apparatus plates are fabricated from aluminum; one laboratory (NPL) utilizes a copper hot plate. The plate emittances range from 0.86 to 0.9, or greater. The plate heaters are either distributed (majority) or line-source [14]. In the case for distributed heaters, two laboratories provided details on different heater constructions (see notes 1 and 3). Edge guarding of the plates was accomplished by lateral insulation, linear guard, environmental chamber, or a combination of these approaches. For temperature measurements, four apparatus utilize thermocouples, either Type E (nickel-chrome/constantan), Type K (nickel-chrome/nickel-silicon), or Type T (copper/constantan). One laboratory (NIST) utilized platinum resistance thermometers (PRTs).

3.2.3 Control Stability: In addition to the information given in Table 4, each laboratory was required to report their criteria to ensure that their data were, in fact, stable (i.e., in a state of statistical control). The responses indicate that each laboratory has developed their own in-house protocol for checking stability of their apparatus during operation. For example, JTCCM requires a temperature difference less than 0.03 K over the last 5 readings taken at 30 min intervals after 24 h steady-state conditions. LNE validates on a 16 h interval from thermal resistance or thermal conductivity measurements using the following criteria: a relative standard deviation 0.05 (%); and, a relative slope of 1.5×10^{-8} (% s^{-1}). NIST requires temperature stability for each plate during the test period to be 0.005 °C, or better. During the same period, the variation in the thermal conductivity data must be less than 0.5 %. NPL requires that the temperature difference be stable to better than 0.03 °C over the last 10 readings taken at 1 h intervals. NRCC requires 12 h of steady-state conditions and for each datum to differ from the mean by no more than the relative (expanded, k = 2) uncertainty estimate for the measurement (1 %).

4 Material Characterization

The major goal of the material characterization was to quantify and, if possible, minimize the density variability among specimens of the reference materials (Table 3) that were distributed to the recipient laboratories. This section analyzes the bulk density and thermal conductivity measurements from the source laboratories and addresses the following questions:

1) What is the level of variability of bulk density for the reference materials? and,
2) What is the effect of the bulk density on thermal conductivity?

A secondary goal of the material characterization is to afford the opportunity for direct comparison of source and recipient laboratory test data. The reader, however, is cautioned that such a comparison is for informational purposes only. Although desirable, other material properties (such as microstructure, mechanical properties, etc.) were not requested.

4.1 Bulk Density (Source Laboratories)

Table 5 summarizes the measurements of specimen mass (m), dimensions (x_1, x_2, L) and bulk density[1] (ρ) reported to NIST by the source laboratories. The last column in Table 5 indicates the recipient laboratory for the specimen pair. The data are grouped by material (1 to 4) and coded using the following number assignments (hereafter in this report): 1) NIST SRM 1451; 2) IRMM-440; 3) JTCCM "candidate"; and, 4) NIST SRM 1453. Within each material group, the data is ranked by density of the specimen pair. For brevity, the laboratories have been assigned a number code (hereafter): 1) JTCCM; 2) LNE; 3) NIST; 4) NPL; and, 5) NRCC.

TABLE 5 – Physical Characteristics of Reference Materials Determined by Source Laboratories

Material	Source Lab	Specimen ID	m (g)	x_1 (mm)	x_2 (mm)	L (mm)	ρ[1] (kg/m^3)	Recipient Lab
1	3	1.1	133.7	611.7	610.0	25.58	14.01	4
1	3	1.2	134.2	613.0	611.0	25.49	14.06	4
1	3	2.1	133.5	610.7	611.7	25.51	14.01	2
1	3	2.2	134.8	611.7	611.3	25.56	14.11	2
1	3	3.1	135.5	611.7	611.3	25.56	14.17	1
1	3	3.2	135.7	612.7	612.0	25.50	14.19	1
1	3	4.1	135.0	611.0	611.3	25.49	14.18	3
1	3	4.2	134.9	610.7	608.3	25.57	14.20	3
1	3	5.1	135.3	609.7	611.0	25.55	14.22	5
1	3	5.2	135.4	609.0	611.0	25.51	14.27	5
2	4	6.1	859	597	598	34.94	68.9	2
2	4	6.2	859	597	596	35.02	69.0	2
2	4	7.1	859	597	596	35.01	68.9	4
2	4	7.2	878	597	598	35.01	70.3	4
2	4	8.1	3624	1199	1202	34.98	71.9	3
2	4	8.2	3683	1198	1201	34.98	73.2	3
2	4	9.1	909	597	596	34.97	73.0	5
2	4	9.2	910	596	598	34.83	73.3	5
2	4	10.1	916	597	599	34.83	73.6	1
2	4	10.2	939	597	596	34.83	75.8	1

[1] In order to preserve the specimens for additional measurements, the source laboratory determined the bulk density of the entire specimen and did not cut the meter section to determine the bulk density for the meter area.

TABLE 5 (continued)

Material	Source Lab	Specimen ID	m (g)	x_1 (mm)	x_2 (mm)	L (mm)	ρ (kg/m^3)	Recipient Lab
3	1	11.1	4482	910	910	24.4	222	1
3	1	11.2	4390	910	910	23.9	222	1
3	1	12.1	4470	910	910	24.3	222	4
3	1	12.2	4413	910	910	23.8	224	4
3	1	13.1	4437	910	910	23.6	227	3
3	1	13.2	4462	910	910	23.7	227	3
3	1	14.1	4456	910	910	23.6	228	2
3	1	14.2	4495	910	910	23.7	229	2
3	1	15.1	4565	910	910	23.7	233	5
3	1	15.2	4501	910	910	23.3	233	5
4	3	16.1	190.6	611.0	611.0	13.48	37.87	4
4	3	16.2	191.5	611.0	611.0	13.52	37.94	4
4	3	17.1	190.3	611.0	610.3	13.42	38.03	5
4	3	17.2	192.4	611.0	611.0	13.50	38.18	5
4	3	18.1	193.7	611.0	611.3	13.60	38.13	3
4	3	18.2	186.4	611.0	611.0	13.08	38.17	3
4	3	19.1	193.2	611.0	610.7	13.46	38.47	1
4	3	19.2	193.4	611.0	610.7	13.46	38.51	1
4	3	20.1	191.8	611.0	610.7	13.30	38.65	2
4	3	20.2	194.5	611.0	611.0	13.48	38.65	2

Figure 1 plots the density measurements given in Table 5 versus specimen pair (1 to 20) for each material. The plot character depicts the recipient laboratory for any given specimen pair. The source laboratories determined the selection of the recipient laboratories for each specimen pair by random lot. The graphs indicate that, for the most part, each pair of specimens was reasonably well matched by bulk density. The grand mean ($\bar{\bar{\rho}}$) for each material is displayed as a solid horizontal line. Values for $\bar{\bar{\rho}}$, the grand standard deviation, and range are summarized in a text box for each material.

Figure 2 plots the relative differences of ρ (Table 5) and $\bar{\bar{\rho}}$ (Figure 1) versus specimen pair for materials 1 through 4. Again, the plot character depicts the recipient laboratory. Figure 2 provides a quick comparison of relative differences for all the materials. For materials 1, 2, 3, and 4, the relative differences range from −1 % to +1%, −4 % to +5.5 %, −2 % to +2.5 %, and −1 % to +1%, respectively (Figure 2). The effect of ρ on λ is investigated in the next section.

Table 6 summarizes the results from Figures 1 and 2.

TABLE 6 – Summary Statistics for Bulk Density Determined by Source Laboratories

Summary Statistic	Material 1	Material 2	Material 3	Material 4
$\bar{\bar{\rho}}$ (kg/m^3)	14.14	71.8	227	38.3
Grand SD (kg/m^3)	0.09	2.4	4.2	0.3
Relative Grand SD (%)	0.6	3.3	1.9	0.8
Range (kg/m^3)	0.26	6.9	11	0.8

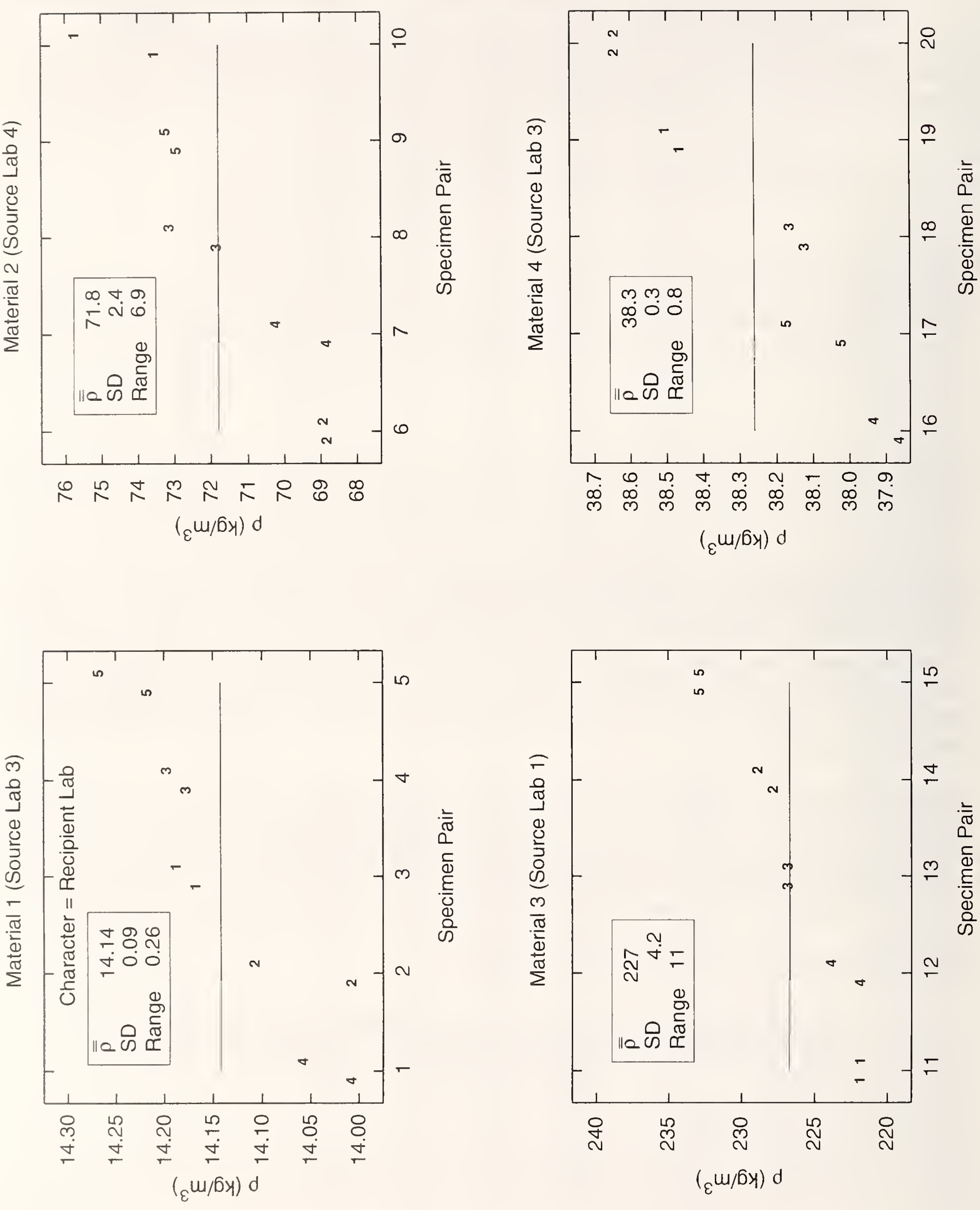


Figure 1. Multi-plot of bulk density versus specimen pair - source laboratory (Materials 1, 2, 3, 4)

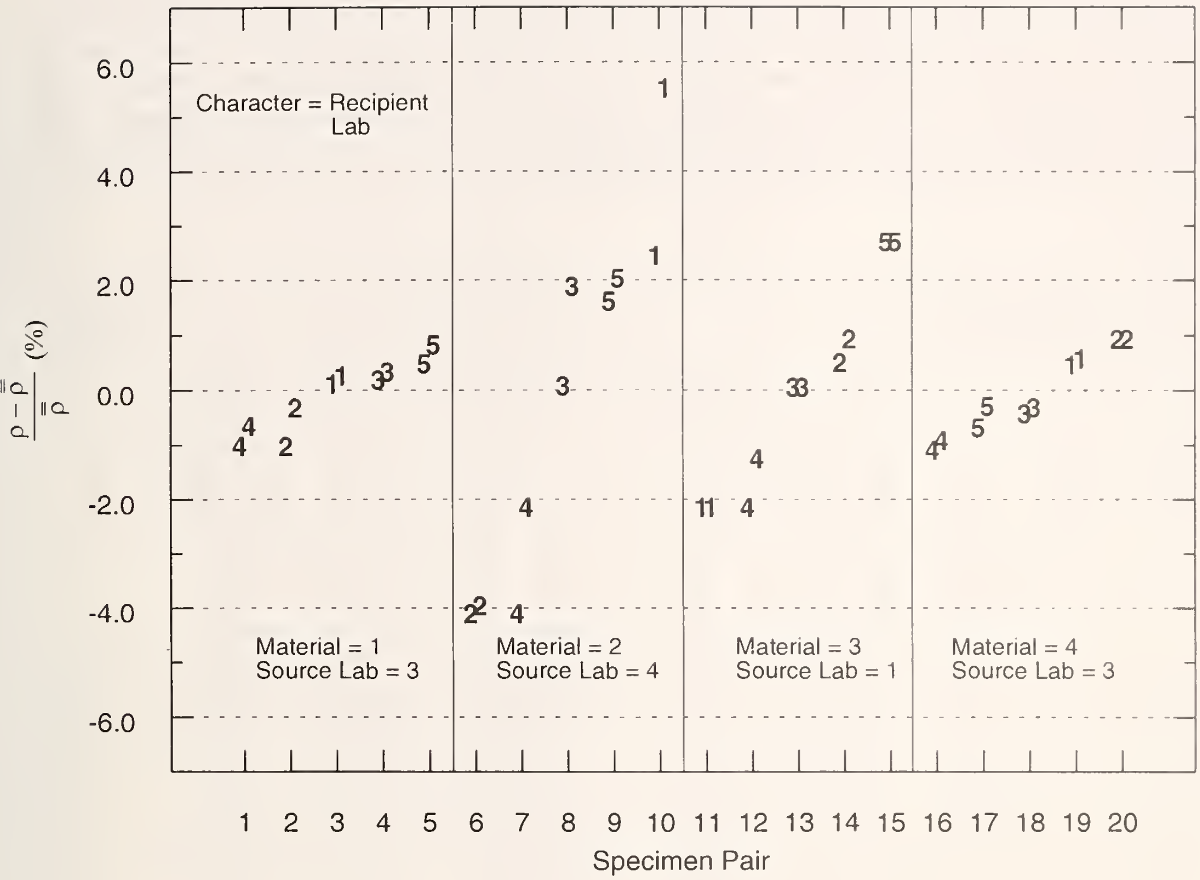


Figure 2. Relative variation in specimen bulk density - source laboratory (Materials 1, 2, 3, 4)

4.2 Thermal Conductivity (Source Laboratories)

Table 7 summarizes measurements reported to NIST by the source laboratories for the hot (T_h) and cold (T_c) plate temperatures, specimen heat flow (Q), thickness (L), meter area (A), and thermal conductivity (λ) at 297.15 K (24 °C) and a temperature difference of 20 K. Ideally, the measurements of λ were determined following equivalent (or similar) procedures used later for the comparison. Hence, laboratory 3 determined the thermal conductivity for each pair of specimens (n = 5) in the double-sided mode of operation and laboratory 4 measured each specimen individually (n = 10) in the single-sided mode. Note that laboratory 1 utilized a 900-mm square guarded hot plate apparatus (450 mm square meter area) to measure each specimen pair individually, which was a different apparatus than that used later in the comparison (Table 4).

TABLE 7 – Thermal Conductivity (297.15 K) of Reference Materials Determined by Source Laboratories

Material	Source Lab	Specimen (Pair)	$\rho^{\dagger}$ (kg/m^3)	T_h (K)	T_c (K)	Q (W)	L (mm)	A (m^2)	λ (W/m K)	Recipient Lab
1	3	1	14.0	307.15	287.15	4.089	25.53	0.1298	0.04021	4
1	3	2	14.1	307.15	287.15	4.152	25.51	0.1298	0.04079	2
1	3	3	14.2	307.15	287.15	4.163	25.53	0.1298	0.04095	1
1	3	4	14.2	307.15	287.15	4.121	25.53	0.1298	0.04053	3
1	3	5	14.2	307.15	287.15	4.087	25.53	0.1298	0.04020	5
2	4	6.1	68.9	305.2	288.1	1.486	34.48	0.09315	0.03212	2
2	4	6.2	69.0	305.0	288.1	1.488	34.42	0.09315	0.03232	2
2	4	7.1	68.9	305.3	288.2	1.482	34.46	0.09315	0.03204	4
2	4	7.2	70.3	305.2	288.1	1.484	34.23	0.09315	0.03206	4
2	4	8.1	71.9	305.3	288.1	1.494	34.43	0.09315	0.03223	3
2	4	8.2	73.2	305.3	288.1	1.493	34.47	0.09315	0.03226	3
2	4	9.1	73.0	305.2	288.1	1.492	34.41	0.09315	0.03218	5
2	4	9.2	73.3	305.1	288.1	1.474	34.42	0.09315	0.0321	5
2	4	10.1	73.6	305.2	288.1	1.488	34.42	0.09315	0.03221	1
2	4	10.2	75.8	304.7	288.1	1.446	34.48	0.09315	0.03216	1
3	1	11.1	222	308.2	287.9	6.413	24.4	0.2025	Not Reported	1
3	1	11.2	222	307.9	287.9	6.269	23.9	0.2025	0.0370	1
3	1	12.1	222	308.1	288.2	6.122	24.3	0.2025	0.0369	4
3	1	12.2	224	308.2	288.1	6.317	23.8	0.2025	0.0369	4
3	1	13.1	227	308.5	288.1	6.474	23.6	0.2025	0.0370	3
3	1	13.2	227	308.2	288.1	6.426	23.7	0.2025	0.0375	3
3	1	14.1	228	307.7	288.1	6.272	23.6	0.2025	0.0373	2
3	1	14.2	229	307.9	288.0	6.351	23.7	0.2025	0.0373	2
3	1	15.1	233	308.5	287.9	6.610	23.7	0.2025	0.0376	5
3	1	15.2	233	308.0	287.9	6.536	23.3	0.2025	0.0374	5
4	3	16	37.9	307.15	287.15	6.489	13.49	0.1298	0.03372	4
4	3	17	38.1	307.15	287.15	6.483	13.46	0.1298	0.03361	5
4	3	18	38.2	307.15	287.15	6.545	13.34	0.1298	0.03363	3
4	3	19	38.5	307.15	287.15	6.466	13.46	0.1298	0.03351	1
4	3	20	38.7	307.15	287.15	6.487	13.39	0.1298	0.03347	2

†Values taken from Table 5.

Figure 3 plots the thermal conductivity (from Table 7) as a function of their respective bulk densities (ρ) (from Table 5) for each material. The plot character depicts the recipient laboratory for the specimens thereby facilitating a direct comparison with later results from the recipient laboratories.

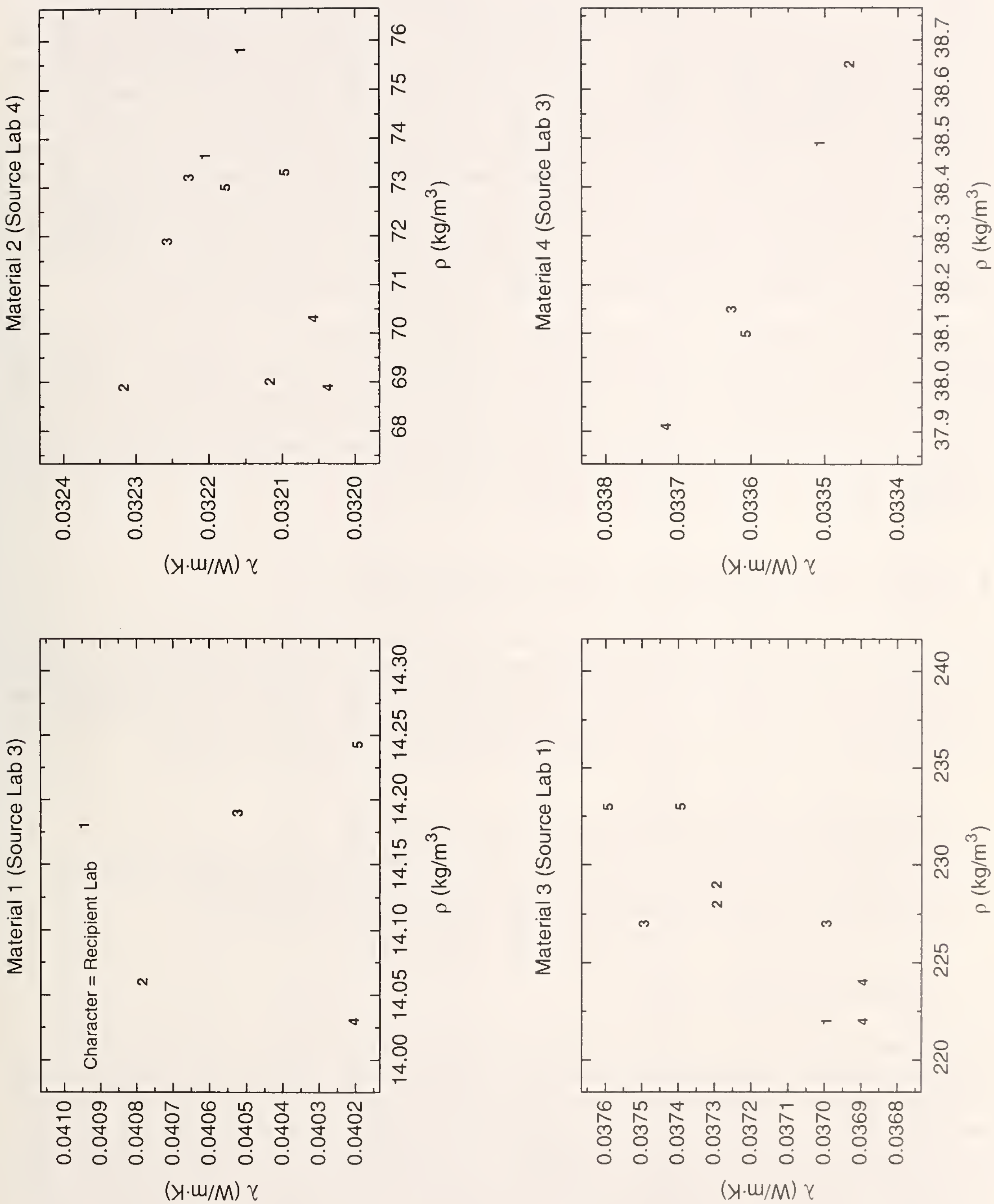


Figure 3. Multi-plot of thermal conductivity versus bulk density - source laboratory (Materials 1, 2, 3, 4)

Figure 3 indicates that, for these particular specimens, the λ-ρ relationship is weak (or negligible) for materials 1 and 2; positive for material 3; and, negative for material 4. For materials 2 and 4, these results are consistent with previous findings from References [10] and [11], respectively. Material 2 has been specifically manufactured such that values of λ are essentially independent of ρ from 283 K to 293 K [10]. For material 4, the negative λ-ρ relationship is consistent with the negative density term [11] given in Table 3. For material 1, however, the certification equation [9] indicates a definitive λ-ρ relationship (Table 3), not evident in Figure 3. One possible explanation is that the meter-area ρ affected the test results for material 1. In general, the results of Figure 3 would be more representative of the measured λ if the meter-area ρ was used, especially if there was significant within-specimen variability for material 1. Significant within-specimen variability could also affect the test results from apparatus having significantly different meter areas (for example, laboratories 1 and 3).

The main goal of characterizing each sample of reference material by a source laboratory was to provide the recipient laboratories with closely equivalent sets of test specimens. Further, by having an issuing laboratory conduct thermal conductivity measurements on each specimen, there is an opportunity for a direct comparison of the source and recipient laboratories. An analysis of these data (for informational purposes only) will be presented in the next section. Finally, it is important to note that the material characterization data were only released to the participants at the conclusion of the test program.

5 Analysis of Fixed Temperature (297.15 K) Replicate Data

This section provides a tabulated listing of the fixed temperature (297.15 K) replicate data and the corresponding statistical analyses of the data. The primary goal of the analyses is to provide the laboratory participants with diagnostic tools for examination of their data. The analyses are motivated by the central theme of the comparison: How do the laboratories behave across the four materials? With respect to this question, the following analyses are provided:

1) Presentation of laboratory data;
2) Graphical exploration of laboratory test data by material;
3) Assessment of the engineering significance of the laboratory test data by material;
4) Detailed statistical analysis of the location and variation of the test data;
5) An ensuing ranking of the laboratories by location and variation; and,
6) Comparison of source and recipient laboratory test data.

5.1 Presentation of Laboratory Data

Table 8 summarizes the measurements for the specimen bulk density (ρ), hot (T_h) and cold (T_c) plate temperatures, specimen heat flow (Q), thickness (L), meter area (A), and thermal conductivity (λ) at 297.15 K (24 °C) and a temperature difference of 20 K. The data in Table 8 were entered in an electronic spreadsheet and report values for λ were checked by recalculation. For convenience, values of ρ have been rounded to 3 significant digits and each value represents the average of the pair of specimens (for a double sided test). The number of significant digits for the other parameters is tabulated (here and elsewhere) as received from the laboratory. Any certainty for the 5th significant digit for values of λ cannot be assigned, but the digit is included for the subsequent analyses of the results.

TABLE 8 – Fixed Temperature (297.15 K) Replicate Data

Material	Lab	Replicate	ρ (kg/m³)	T_h (K)	T_c (K)	Q (W)	L (mm)	A (m²)	λ (W/m K)	T_m (K)	ΔT (K)
1	1	1	13.4	307.20	286.73	1.622	25.4	0.0225	0.04473	296.97	20.47
1	1	2	13.4	307.01	286.74	1.612	25.4	0.0225	0.04489	296.88	20.27
1	1	3	13.4	303.16	286.59	1.299	25.4	0.0225	0.04425	294.88	16.57
1	1	4	13.4	303.23	286.54	1.306	25.4	0.0225	0.04417	294.89	16.69
1	1	5	13.4	307.20	286.54	1.623	25.4	0.0225	0.04434	296.87	20.66
1	2	1	14.1	307.14	287.18	5.806	25.40	0.090085	0.04101	297.16	19.96
1	2	2	14.1	307.17	287.16	5.824	25.40	0.090085	0.04104	297.17	20.01
1	2	3	14.1	307.19	287.18	5.824	25.40	0.090085	0.04105	297.19	20.01
1	2	4	14.1	307.20	287.20	5.824	25.40	0.090085	0.04103	297.20	20.00
1	2	5	14.1	307.19	287.21	5.824	25.40	0.090085	0.04109	297.20	19.98
1	3	1	14.2	307.15	287.15	4.124	25.51	0.1298	0.04052	297.15	20.00
1	3	2	14.2	307.15	287.15	4.126	25.52	0.1298	0.04055	297.15	20.00
1	3	3	14.2	307.15	287.15	4.126	25.52	0.1298	0.04056	297.15	20.00
1	3	4	14.2	307.15	287.15	4.127	25.52	0.1298	0.04056	297.15	20.00
1	3	5	14.2	307.15	287.15	4.126	25.52	0.1298	0.04056	297.15	20.00
1	4	1	14.1	306.47	287.54	2.8766	25.42	0.09315	0.0415	297.01	18.93
1	4	2	14.1	306.63	287.57	2.8770	25.37	0.09315	0.0411	297.10	19.06
1	4	3	14.1	306.65	287.58	2.8793	25.37	0.09315	0.0411	297.12	19.07
1	4	4	14.1	306.64	287.57	2.8765	25.37	0.09315	0.0411	297.11	19.07
1	4	5	14.1	306.60	287.57	2.8714	25.37	0.09315	0.0411	297.09	19.03
1	5	1	14.4	307.15	287.17	1.988	25.35	0.0625	0.04035	297.16	19.98
1	5	2	14.4	307.16	287.14	1.987	25.39	0.0625	0.04030	297.15	20.02
1	5	3	14.4	307.14	287.13	1.985	25.39	0.0625	0.04029	297.14	20.01
1	5	4	14.4	307.12	287.16	1.981	25.39	0.0625	0.04031	297.14	19.96
1	5	5	14.4	307.12	287.15	1.985	25.36	0.0625	0.04034	297.14	19.97
2	1	1	79.2	307.21	286.22	0.891	34.5	0.0225	0.03254	296.72	20.99
2	1	2	79.2	307.12	286.20	0.885	34.5	0.0225	0.03243	296.66	20.92
2	1	3	79.2	307.45	286.27	0.899	34.5	0.0225	0.03254	296.86	21.18
2	1	4	79.2	307.47	286.25	0.899	34.5	0.0225	0.03248	296.86	21.22
2	1	5	79.2	307.47	286.27	0.900	34.5	0.0225	0.03255	296.87	21.20
2	2	1	69.9	307.15	287.20	3.329	34.41	0.090085	0.03186	297.18	19.95
2	2	2	69.9	307.17	287.17	3.340	34.41	0.090085	0.03189	297.17	20.00
2	2	3	69.9	307.20	287.12	3.351	34.41	0.090085	0.03188	297.16	20.08
2	2	4	69.9	307.20	287.14	3.351	34.41	0.090085	0.03189	297.17	20.06
2	2	5	69.9	307.24	287.25	3.339	34.41	0.090085	0.03191	297.25	19.99
2	3	1	73.8	307.15	287.15	2.356	34.83	0.1298	0.03161	297.15	20.00
2	3	2	73.8	307.15	287.15	2.362	34.82	0.1298	0.03168	297.15	20.00
2	3	3	73.8	307.15	287.14	2.362	34.82	0.1298	0.03167	297.15	20.01
2	3	4	73.8	307.15	287.14	2.365	34.82	0.1298	0.03170	297.15	20.01
2	3	5	73.8	307.15	287.15	2.360	34.82	0.1298	0.03164	297.15	20.00
2	4	1	70.0	306.16	287.30	1.6364	34.38	0.09315	0.0320	296.73	18.86
2	4	2	70.0	306.15	287.31	1.6379	34.34	0.09315	0.0321	296.73	18.84
2	4	3	70.0	306.19	287.31	1.6435	34.34	0.09315	0.0321	296.75	18.88
2	4	4	70.0	306.16	287.31	1.6358	34.35	0.09315	0.0320	296.74	18.85
2	4	5	70.0	306.09	287.29	1.6380	34.33	0.09315	0.0321	296.69	18.80
2	5	1	74.6	307.04	287.14	1.161	34.14	0.0625	0.03188	297.09	19.90
2	5	2	74.6	307.17	287.14	1.182	34.14	0.0625	0.03223	297.16	20.03
2	5	3	74.6	307.14	287.15	1.182	34.14	0.0625	0.03232	297.15	19.99
2	5	4	74.6	307.15	287.17	1.181	34.14	0.0625	0.03229	297.16	19.98
2	5	5	74.6	307.15	287.17	1.181	34.14	0.0625	0.03228	297.16	19.98

Material	Lab	Replicate	ρ (kg/m³)	T_h (K)	T_c (K)	Q (W)	L (mm)	A (m²)	λ (W/m K)	T_m (K)	ΔT (K)
3	1	1	213	307.09	286.36	1.328	25.3	0.0225	0.03602	296.73	20.73
3	1	2	213	307.32	286.36	1.360	25.3	0.0225	0.03648	296.84	20.96
3	1	3	213	307.48	286.35	1.363	25.3	0.0225	0.03627	296.92	21.13
3	1	4	213	306.83	286.31	1.352	25.3	0.0225	0.03704	296.57	20.52
3	1	5	213	306.90	286.34	1.351	25.3	0.0225	0.03694	296.62	20.56
3	2	1	228	307.17	287.26	5.611	23.48	0.090085	0.03674	297.22	19.91
3	2	2	228	307.21	287.36	5.596	23.48	0.090085	0.03673	297.29	19.85
3	2	3	228	307.18	287.32	5.610	23.44	0.090085	0.03677	297.25	19.86
3	2	4	228	307.22	287.26	5.640	23.45	0.090085	0.03676	297.24	19.96
3	2	5	228	307.12	287.25	5.609	23.45	0.090084	0.03675	297.19	19.87
3	3	1	225	307.15	287.15	3.958	23.73	0.1298	0.03617	297.15	20.00
3	3	2	225	307.15	287.15	3.921	23.76	0.1298	0.03588	297.15	20.00
3	3	3	225	307.15	287.15	3.949	23.88	0.1298	0.03633	297.15	20.00
3	3	4	225	307.15	287.15	3.952	23.77	0.1298	0.03618	297.15	20.00
3	3	5	225	307.15	287.15	3.962	23.76	0.1298	0.03625	297.15	20.00
3	4	1	223	306.26	287.48	2.5446	24.10	0.09315	0.0350	296.87	18.78
3	4	2	223	306.23	287.47	2.5399	24.09	0.09315	0.0350	296.85	18.76
3	4	3	223	306.27	287.46	2.5442	24.11	0.09315	0.0350	296.87	18.81
3	4	4	223	306.25	287.48	2.5403	24.11	0.09315	0.0350	296.87	18.77
3	4	5	223	306.19	287.46	2.5352	24.09	0.09315	0.0350	296.83	18.73
3	5	1	230	307.14	287.14	1.963	23.48	0.0625	0.03688	297.14	20.00
3	5	2	230	307.14	287.14	1.965	23.48	0.0625	0.03691	297.14	20.00
3	5	3	230	307.13	287.15	1.960	23.48	0.0625	0.03687	297.14	19.98
3	5	4	230	307.15	287.13	1.963	23.48	0.0625	0.03685	297.14	20.02
3	5	5	230	307.17	287.15	1.961	23.48	0.0625	0.03680	297.16	20.02
4	1	1	39.8	306.50	285.80	2.347	13.5	0.0225	0.03401	296.15	20.70
4	1	2	39.8	306.92	286.41	2.347	13.5	0.0225	0.03433	296.67	20.51
4	1	3	39.8	306.21	285.19	2.347	13.5	0.0225	0.03350	295.70	21.02
4	1	4	39.8	306.38	285.56	2.347	13.5	0.0225	0.03382	295.97	20.82
4	1	5	39.8	306.57	285.80	2.347	13.5	0.0225	0.03390	296.19	20.77
4	2	1	38.6	307.15	287.15	9.046	13.43	0.090085	0.03369	297.15	20.00
4	2	2	38.6	307.16	287.11	9.065	13.44	0.090085	0.03369	297.14	20.05
4	2	3	38.6	307.14	287.18	9.028	13.43	0.090085	0.03372	297.16	19.96
4	2	4	38.6	307.14	287.13	9.045	13.42	0.090085	0.03368	297.14	20.01
4	2	5	38.6	307.20	287.16	9.065	13.42	0.090085	0.03368	297.18	20.04
4	3	1	38.2	307.15	287.15	6.557	13.33	0.1298	0.03367	297.15	20.00
4	3	2	38.2	307.15	287.15	6.578	13.33	0.1298	0.03378	297.15	20.00
4	3	3	38.2	307.15	287.15	6.576	13.33	0.1298	0.03377	297.15	20.00
4	3	4	38.2	307.15	287.15	6.575	13.33	0.1298	0.03376	297.15	20.00
4	3	5	38.2	307.15	287.15	6.578	13.32	0.1298	0.03377	297.15	20.00
4	4	1	38.8	305.66	287.90	4.2130	13.17	0.09315	0.0335	296.78	17.76
4	4	2	38.8	305.69	287.87	4.2328	13.25	0.09315	0.0338	296.78	17.82
4	4	3	38.8	305.77	287.90	4.2410	13.26	0.09315	0.0338	296.84	17.87
4	4	4	38.8	305.69	287.89	4.2290	13.20	0.09315	0.0337	296.79	17.80
4	4	5	38.8	305.67	287.88	4.2296	13.14	0.09315	0.0336	296.78	17.79
4	5	1	38.5	307.12	287.13	3.168	13.36	0.0625	0.03387	297.13	19.99
4	5	2	38.5	307.13	287.16	3.169	13.34	0.0625	0.03386	297.15	19.97
4	5	3	38.5	307.12	287.14	3.170	13.34	0.0625	0.03386	297.13	19.98
4	5	4	38.5	307.13	287.14	3.171	13.35	0.0625	0.03389	297.14	19.99
4	5	5	38.5	307.13	287.13	3.172	13.35	0.0625	0.03388	297.13	20.00

5.2 Graphical Exploration of Laboratory Data

Figure 4 plots the measurements of λ at 297.15 K versus laboratory (1 to 5) for the 4 materials. The replicate observations for each laboratory, shown as × characters, are slightly offset along the x-axis to assess any trends in the run-sequence of an individual laboratory. In examining the laboratory data for each material, the central question under investigation is, do the five laboratories behave similarly across the four materials? or, (if the laboratories behave differently from material to material), is there a laboratory-material interaction? There are two independent but related questions in determining the behavior of laboratories from material to material: 1) Is there a change in location (that is, mathematical mean) of the laboratory data? and, 2) Is there a change in variation (standard deviations) of the laboratory data? Examination of these two questions for the data in Figure 4 will provide an answer for the central question under investigation (i.e., How do the five laboratories behave across the four materials?).

The principal conclusion from Figure 4 is that the behavior of the laboratories does, in fact, change from material to material. As observed in the plots, the location and variation of each set of laboratory data changes from material to material. For example, laboratory 5 is lowest and relatively precise for material 1, and higher with respect to the other laboratories and less precise for material 2. In short, there is a laboratory-material interaction. Since the breadth of this interaction was not entirely anticipated to the extent observed in the data (for example, material 1, laboratory 1), the most immediate response is, why? What are the principal causes for the laboratory-material interaction? A helpful plan of attack is to refine the problem by focusing attention on the extreme values of the data set with respect to location and variation. For each material, Table 9 summarizes the laboratories with the highest and lowest mean values, the highest variation (i.e., noise), and apparent outliers.

TABLE 9 – Summary of the Interlaboratory Comparison

Material	High Lab	Low Lab	Noisy Lab	Outlying Lab
1	1	5	1	---
2	1	3	---	5 (1 observation)
3	---	4	1	---
4	---	---	1	---

From Table 9, the major results from the interlaboratory comparison are summarized below as a set of primary questions.

1) Q1: For 2 of the 4 materials, laboratory 1 is high, why?
2) Q2: For material 1, laboratory 5 is low, why?
3) Q3: For material 2, laboratory 3 is low, why?
4) Q4: For material 3, laboratory 4 is low, why?
5) Q5: For 3 of the 4 materials, laboratory 1 is noisy, why?
6) Q6: For material 2, laboratory 5 appears to have one outlying observation, why?

The answers to this set of questions are of considerable interest and will guide the presentation of more detailed analyses presented later in this report (Section 6). These analyses investigate the effects of material factors (including bulk density, etc.) as well as other factors on λ. Before proceeding with these detailed analyses, however, it is extremely useful to investigate the engineering significance of the test data given in Figure 4.

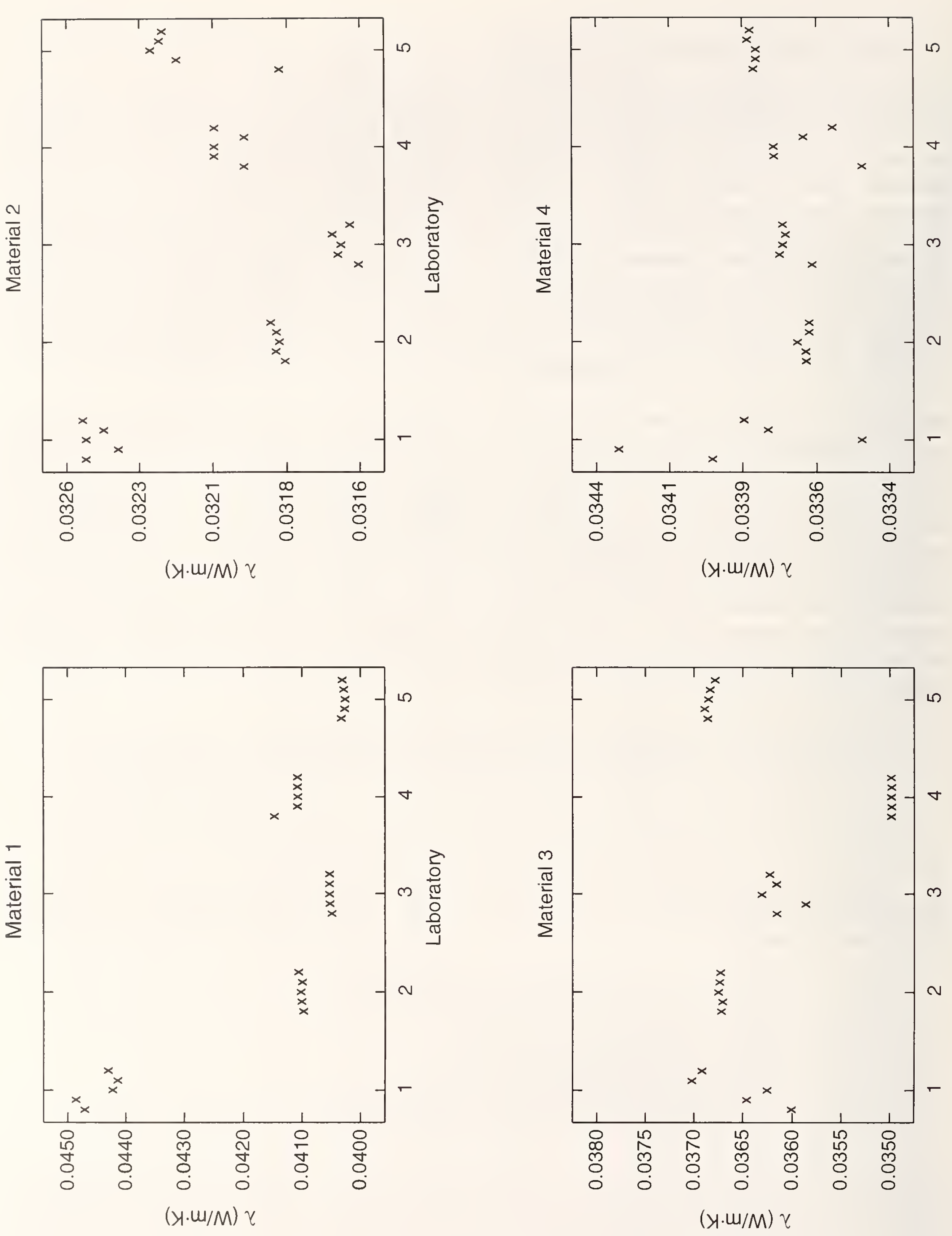


Figure 4. Replicate data (297.15 K) versus laboratory (Materials 1, 2, 3, 4)

5.3 Engineering Significance of Laboratory Data

Based on the results of Figure 4, an appropriate question is, are the differences in the laboratory test data significant from an engineering viewpoint? In other words, are the differences less than some acceptable minimum level of engineering significance? An engineering viewpoint requires some assessment of the uncertainty for laboratory values of λ. From Table 4, recall that for laboratories 2, 3, 4, and 5 the estimates for their relative expanded uncertainties, based on current international guidelines [13], were 1.5 %, 1.5 % for material 2 and 1.0 % for the other materials, 1.2 %, and 1.0 %, respectively. (Laboratory 1 did not report an uncertainty for their values of λ.) For comparison purposes, the relative expanded uncertainties (U) given in Table 4 are presented as symmetric error bars for the data given in Figure 4. It is important to note that the estimates for U reported by each laboratory have been determined independently of this comparison.

Figure 5 again plots the measurements of λ at 297.15 K, shown as × characters, versus laboratory (1 to 5) for each material. As before, the replicate observations for a given laboratory are slightly offset along the x-axis. For laboratories 2, 3, 4, and 5, the data points include symmetric error bars representing the respective laboratory's estimate of $U(\lambda)$. As stated earlier, U defines an interval about the result of a measurement that may be expected to encompass a large fraction of the distribution of values that could easily be attributed to the measurand [13] (i.e., λ). For a coverage factor of $k = 2$, U defines a 95 % level of confidence within which the value of thermal conductivity is confidently believed to reside [13].

Examination of the data in Figure 5 confirms the primary findings from the graphical analysis shown in Figure 4. That is, there are systematic differences among the laboratory data for materials 1, 2, and 3. For these materials there are several cases where the differences among the data points lie outside the ranges covered by the error bars, most notably for materials 1 and 3. In contrast, however, the differences for material 4 among laboratories 2, 3, 4, 5, and 4 of the 5 observations for laboratory 1 appear to be insignificant at the uncertainty levels given in Table 4. Also, on a positive note, almost all the individual variation noted within a laboratory lies within the error bars given for the laboratory. An exception is noted for material 2, laboratory 5.

The evaluation of sources for the systematic differences (effects due to differences in specimens, equipment, procedure, etc.) noted in Figure 5 is described in Section 6. Further diagnosis of the laboratory replicate data, including a detailed investigation of the location (means) and variation (standard deviations) for the data, is summarized below.

5.4 Detailed Investigation of Laboratory Location and Variation

The general conclusions from Figures 4 and 5 are examined further by the application of the mean and standard deviation plots to the thermal conductivity data. The mean plot provides quantitative information for the question: Is there a change in the laboratory means across laboratories? Likewise, the standard deviation plot provides quantitative information for the question: Is there a change in the laboratory variation across laboratories? In the first case, the change in location of the mean is associated with a systematic difference between laboratories. For the second case, independent replicate measurements are generally used to provide estimates of within- (and also between-) laboratory precision. The analyses of these statistics will be presented graphically, tabulated, and used to rank the laboratories.

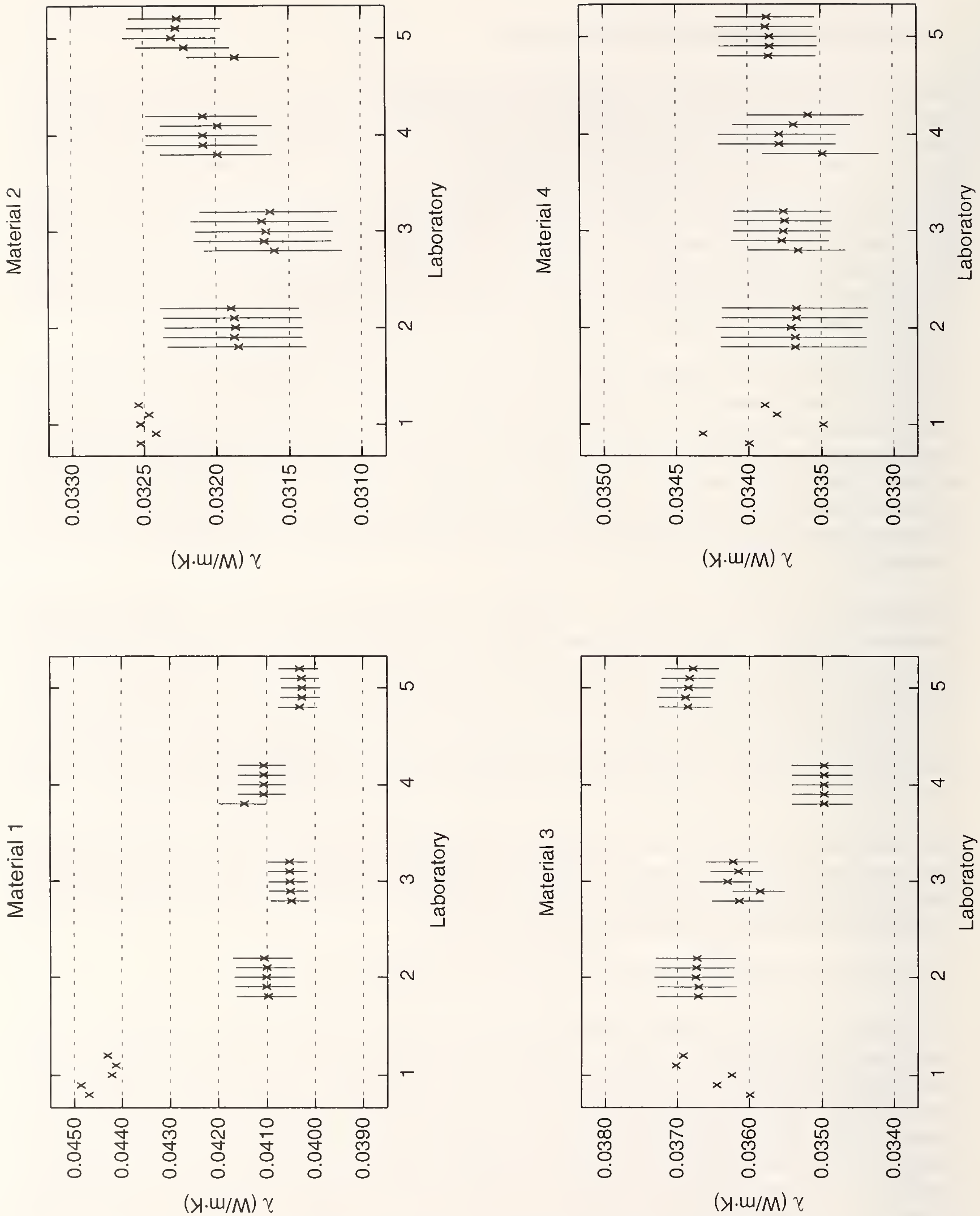


Figure 5. Replicate data (297.15 K) versus laboratory (Materials 1, 2, 3, 4)
Error bars equal laboratory relative expanded uncertainty (Table 4)

Figure 6 plots the mean ($\bar{\lambda}$) value for each laboratory (where $n = 5$ observations) for materials 1 through 4. The grand mean ($\bar{\bar{\lambda}}$) for each material ($n = 25$ observations) is shown as a horizontal line. For reference, summary statistics that include $\bar{\bar{\lambda}}$, grand standard deviation, and range are provided in a text box for each material. Ideally, the differences between $\bar{\lambda}$ and $\bar{\bar{\lambda}}$ should be small, indicating close agreement among the laboratories, and randomly scattered about the horizontal line. An examination of the plots reveals that the locations of the means (with respect to the laboratories) change from material to material. Ranking the materials by the magnitude of their differences (by using either the grand standard deviation or range) reveals the following order (from highest to smallest): 1, 3, 2, and 4.

Figure 7 plots the standard deviation for each laboratory ($n = 5$ observations) for materials 1 through 4. Here, each point represents the within-laboratory variability for five (independent) observations, which, in most cases, is quite small. The grand standard deviation for each material ($n = 25$ observations), shown as a solid horizontal line, includes both within- and between-laboratory variability. An examination of the plots reveals that the variability within a laboratory also changes from material to material. It is interesting to note that for materials 1, 2, and 3, the grand standard deviation is larger than the laboratory standard deviations and, for material 4, is comparable to the laboratory standard deviations.

Tables 10a and 10b summarize the results of Figures 6 and 7, respectively. The last row in each table provides the respective grand or "pooled" statistic ($n = 25$ observations) for each material (across all laboratories). Likewise, the last column in each table provides the respective grand or "pooled" statistic ($n = 25$ observations) for each laboratory (across all materials). As a note of clarification, the pooled standard deviation for each material in Table 10b is determined differently than the grand standard deviation presented in Figures 6 and 7. The results given Table 10 provide quantitative verification of the core conclusions given in Table 9.

TABLE 10a – Means for Replicate Data (297.15 K)

Lab	Material 1	Material 2	Material 3	Material 4	Lab
	$\bar{\lambda}$ (W/m K)	$\bar{\lambda}$ (W/m K)	$\bar{\lambda}$ (W/m K)	$\bar{\lambda}$ (W/m K)	Average (W/m K)
1	0.04448	0.03251	0.03655	0.03391	0.03686
2	0.04104	0.03189	0.03675	0.03369	0.03584
3	0.04055	0.03166	0.03616	0.03375	0.03553
4	0.04118	0.03206	0.03500	0.03368	0.03548
5	0.04032	0.03220	0.03686	0.03387	0.03581
Grand	0.04151	0.03206	0.03626	0.03378	0.03591

TABLE 10b – Standard Deviations for Replicate Data (297.15 K)

Lab	Material 1	Material 2	Material 3	Material 4	Pooled
	SD (λ) (W/m K)	SD (λ) (W/m K)	SD (λ) (W/m K)	SD (λ) (W/m K)	SD (W/m K)
1	0.00032	0.00005	0.00043	0.00030	0.00031
2	0.00003	0.00002	0.00002	0.00002	0.00002
3	0.00002	0.00004	0.00017	0.00005	0.00009
4	0.00018	0.00005	0.00000	0.00013	0.00011
5	0.00003	0.00018	0.00004	0.00001	0.00009
Pooled	0.00016	0.00009	0.00021	0.00015	0.00016

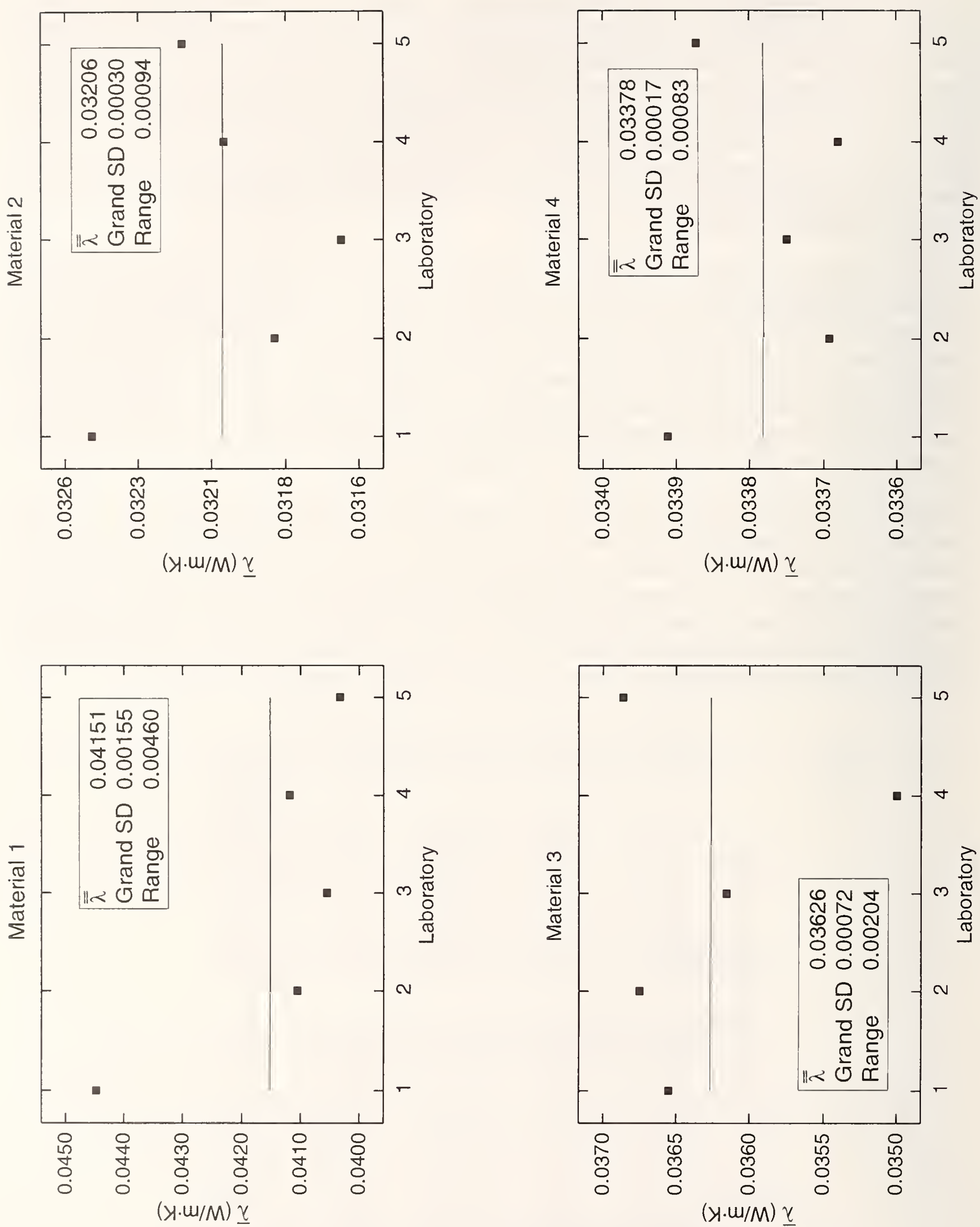


Figure 6. Mean plot of replicate data (297.15 K) versus laboratory (Materials 1, 2, 3, 4)

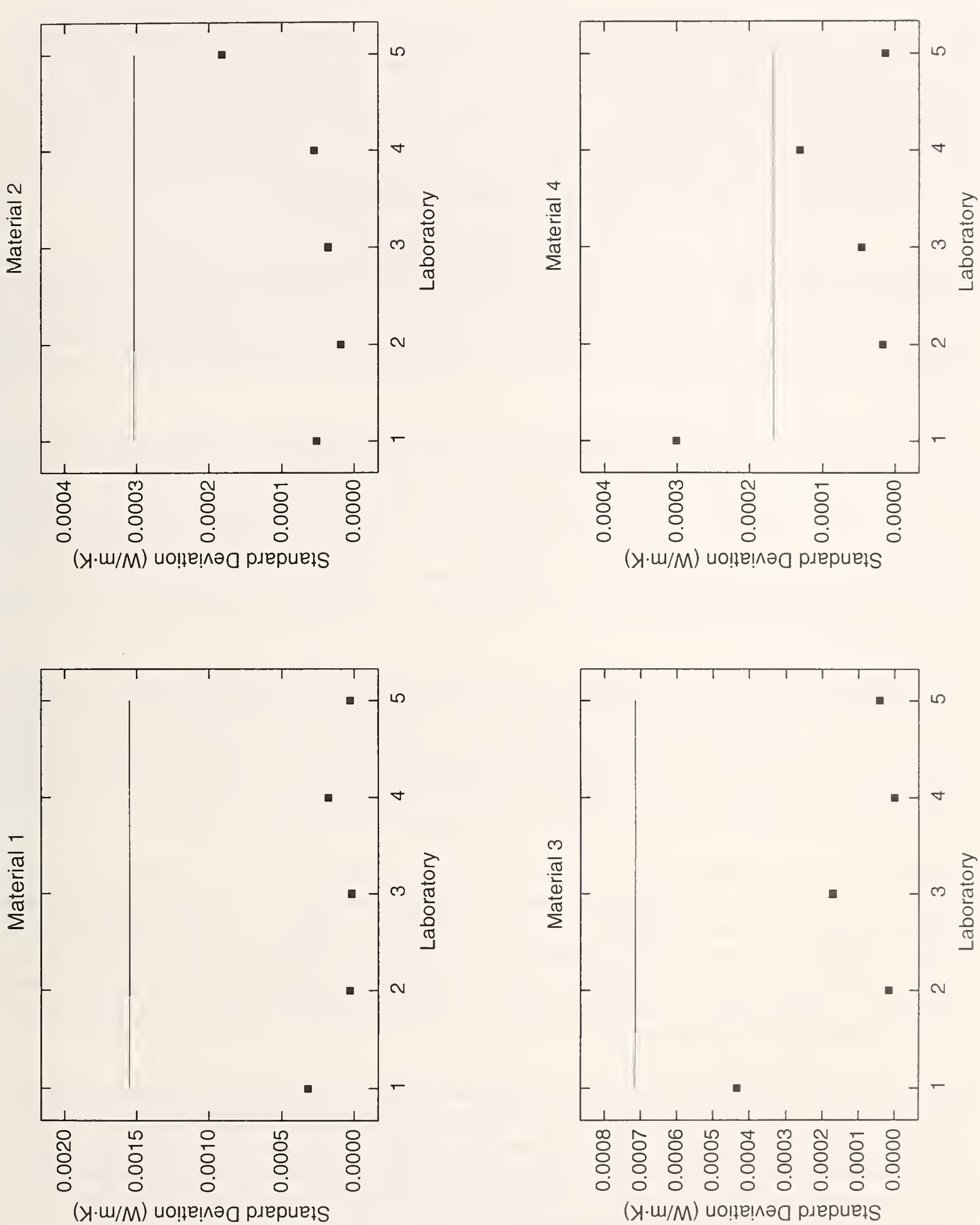


Figure 7. Standard deviation plot of replicate data (297.15 K) versus laboratory (Materials 1, 2, 3, 4)

With regard to location, the last column in Table 10a (lab average across all four materials) indicates that laboratory 1 is consistently higher than the other laboratories. On the average across all four materials, laboratories 2 and 5, and 3 and 4, are closely paired and each pair of laboratories differs by about 0.8%. With regard to variation, the last column in Table 10b indicates that laboratory 1 is consistently noisy across all four materials. Laboratories 3, 4, and 5 exhibit similar levels of variability while laboratory 2 is extremely precise (by nearly a factor of 5 in comparison to the other three laboratories) across all four materials.

The summary statistics given in the last row of Tables 10a and 10b (as well as Figures 6 and 7) motivate the following question of interest: Does the measurement precision vary with the thermal conductivity of the materials being measured? To answer this question, we plot the imprecision for each material and include both within-laboratory variability (pooled standard deviations) and within- and between-laboratory variability (grand standard deviation) versus the grand means of each material.

Figure 8 plots both the pooled (within-laboratory) and grand standard deviations (within- and between-laboratory) versus the grand means of the thermal conductivities for materials 1 through 4. Each data point is depicted by a plot character equal to material. The results of Figure 8 would seem to indicate that the between-laboratory variability increases with the thermal conductivity of the materials. As will be shown later in Figure 24, this trend is an artifact of extremely high and low sets of data for materials 1 and 3, respectively, (as summarized in Table 9). The within-laboratory variability, however, is approximately the same across all levels of thermal conductivity.

For comparison purposes, it is useful to determine the mean and standard deviation statistics (Figures 6 and 7, respectively) on a relative basis (%). Figure 9 plots the relative mean for each laboratory (n = 5 observations) for materials 1 through 4. The relative mean is defined as the percentage difference between $\bar{\lambda}$ (lab mean) and $\bar{\bar{\lambda}}$ (grand mean). With the exception of the extremely high and low means for materials 1 (laboratory 1) and 3 (laboratory 4), respectively, most of the laboratory means are within 1.5 % of the grand mean for each material.

Figure 10 plots the relative standard deviations for each laboratory (n = 5 observations) for materials 1 through 4. Each point represents the within-laboratory variability and the grand standard deviation for each material (n = 25 observations), shown as a solid horizontal line, includes both within- and between-laboratory variability. With the exception of one point (material 3, laboratory 1) all of the relative laboratory standard deviations are less than 1 % and several are less than 0.5 %. The grand relative standard deviations range from approximately 0.5 % (material 4) to 3.7 % (material 1).

Tables 11a and 11b summarize the results of Figures 9 and 10, respectively. The last row in each table provides the respective relative range (maximum minus minimum relative mean) or pooled statistic (n = 25 measurements) for each material across all laboratories. Likewise, the last column in each table provides the respective grand or "pooled" statistic (n = 25 observations) for each laboratory across all materials. In both tables, curious (or anomalous) results for a particular laboratory, relative to the other laboratories, are tagged with a superscript number.

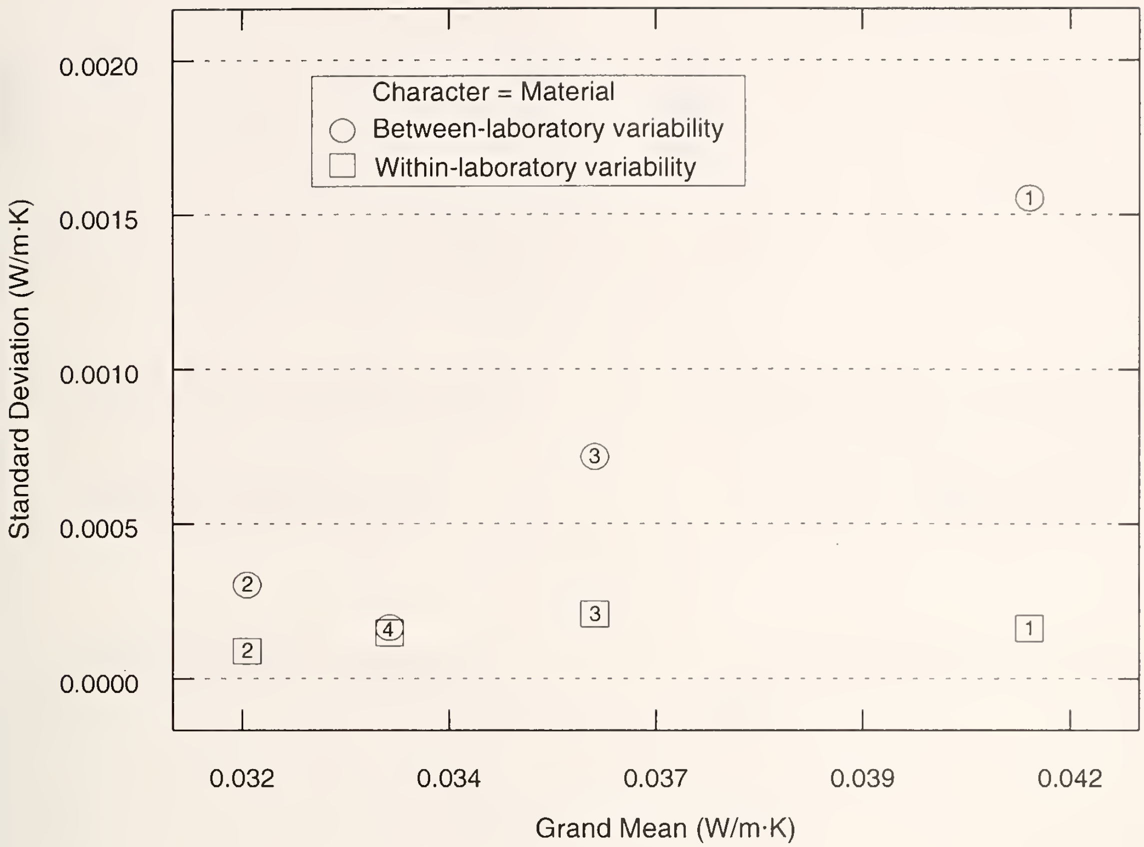


Figure 8. Within- and between-laboratory variability versus grand means (Materials 1, 2, 3, 4)

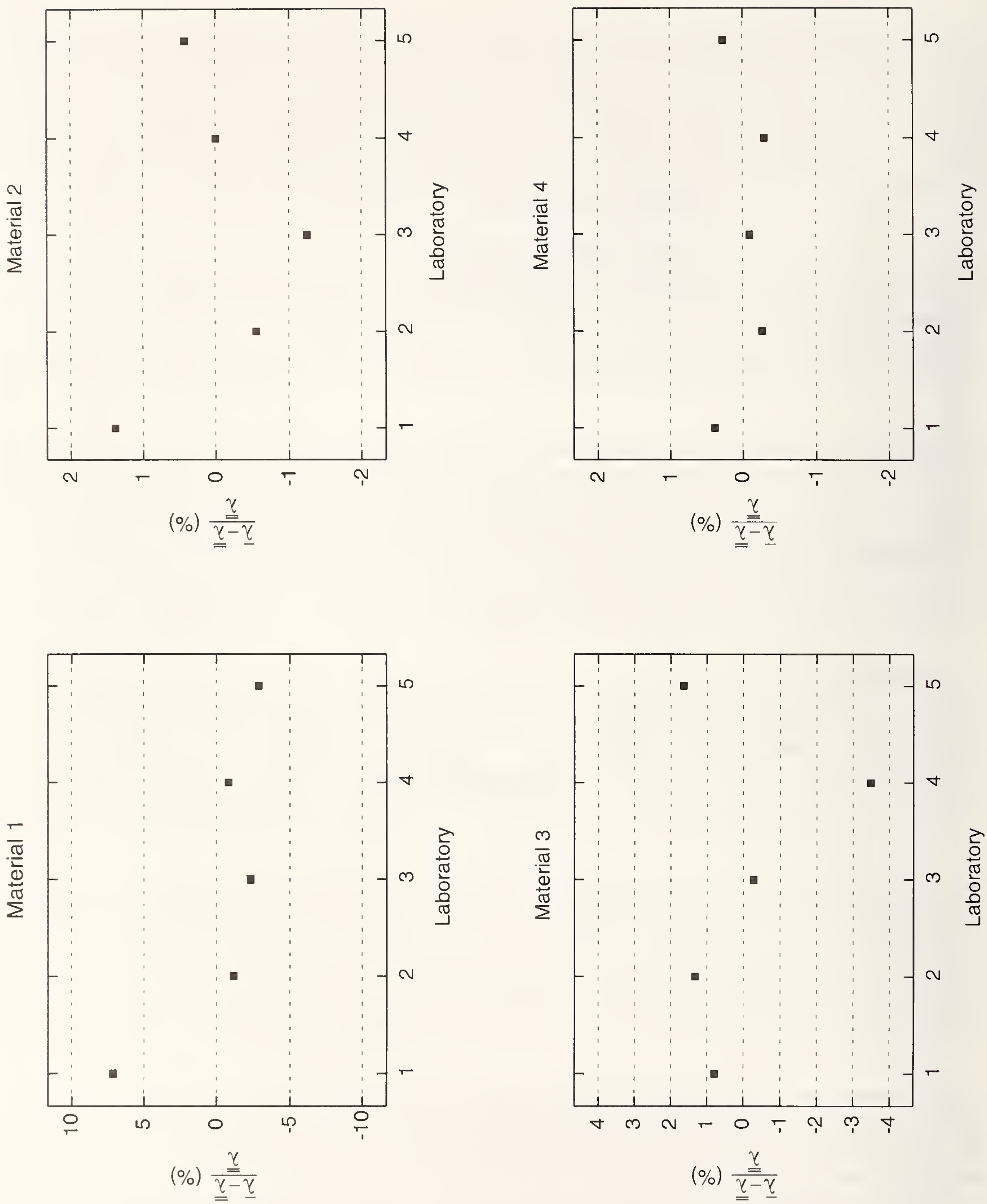


Figure 9. Relative mean plot of replicate data (297.15 K) versus laboratory (Materials 1, 2, 3, 4)

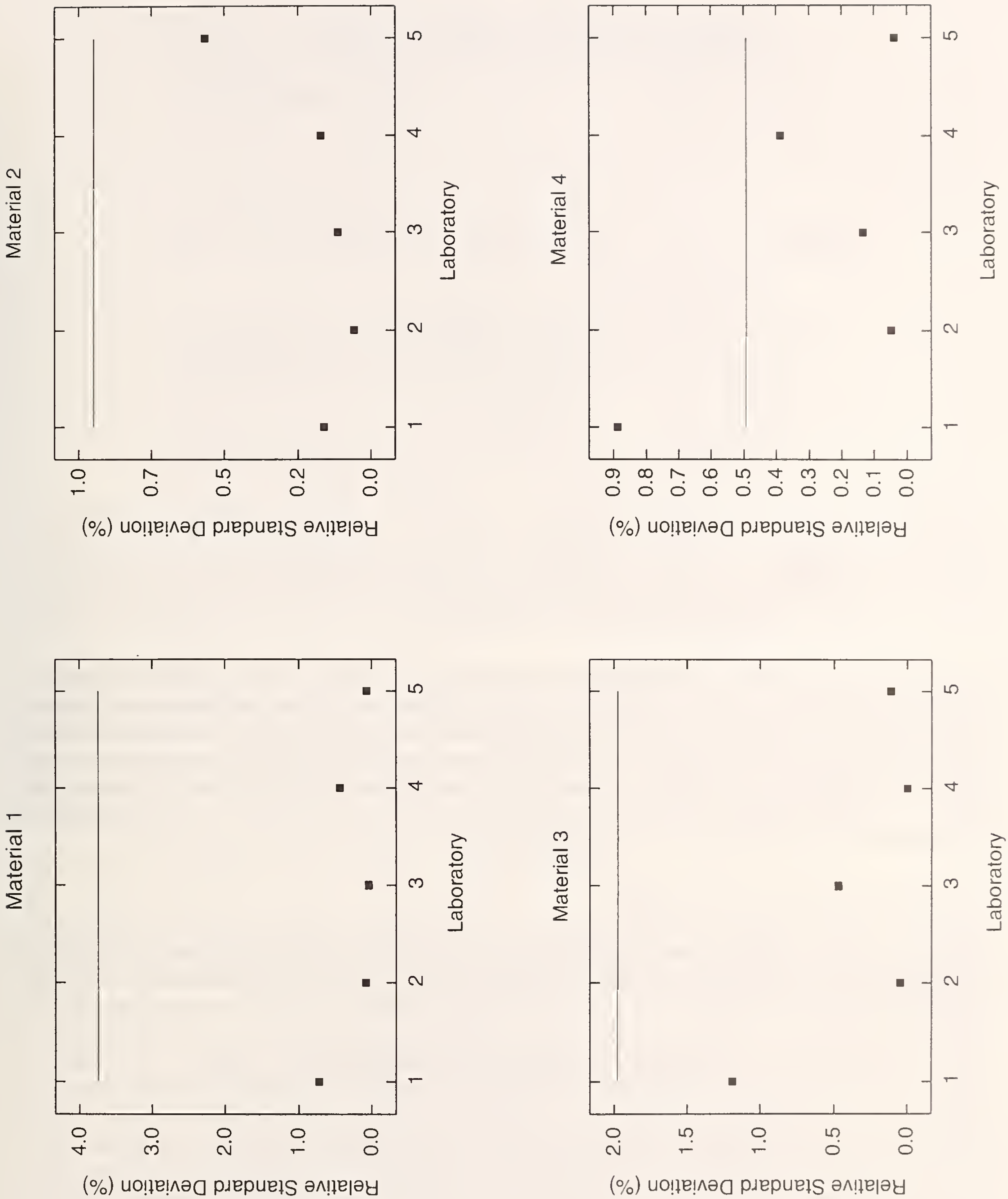


Figure 10. Relative standard deviation plot of replicate data (297.15 K) versus laboratory (Materials 1, 2, 3, 4)

TABLE 11a – Relative Means for Replicate Data (297.15 K)

Lab	Material 1 Mean (%)	Material 2 Mean (%)	Material 3 Mean (%)	Material 4 Mean (%)	Lab Average (%)
1	7.1[1]	1.4	0.8	0.39	2.4
2	−1.1	−0.6	1.3	−0.26	−0.2
3	−2.3	−1.3	−0.3	−0.09	−1.0
4	−0.8	0.0	−3.5[2]	−0.30	−1.2
5	−2.9	0.4	1.6	0.27	−0.2
Range	10.0	2.7	5.1	0.69	3.6

[1]High; [2]Marginally low

TABLE 11b – Relative Standard Deviations for Replicate Data (297.15 K)

Lab	Material 1 SD (%)	Material 2 SD (%)	Material 3 SD (%)	Material 4 SD (%)	Pooled SD (%)
1	0.71[1]	0.16	1.19[1]	0.89[1]	0.83
2	0.07	0.06	0.04	0.05	0.06
3	0.04	0.11	0.47[2]	0.13	0.25
4	0.43[2]	0.17	0.00[3]	0.39[2]	0.30
5	0.06	0.56[1]	0.11	0.04	0.29
Pooled	0.37	0.28	0.57	0.44	---
Grand	3.74	0.95	1.97	0.49	---

[1]High; [2]Marginally high; [3]Artificially low

In general, the treatment of anomalous (or outlying) data can be handled either by retaining, correcting, or deleting the data. Obviously, none of these options are completely satisfactory; however, the third option (deletion) is acceptable when a physical cause can be identified to explain the behavior of the data. As will be seen later, some of the above test data will eventually be classified as outlying (and excluded), but only because of a physical or engineering reason. For these diagnostic analyses, the inclusion of all test data (curious or otherwise) allows the opportunity to rank laboratories as presented below.

5.5 Laboratory and Material Rankings

Using the results of Tables 10 and 11, the laboratory and material rank by location and variation are summarized in Tables 12a, 12b, and 12c, respectively. Note that the rankings given in Tables 12a and 12b verify (and augment) the core conclusions given in Table 9.

TABLE 12a – Laboratory Ranking by Location

Rank	Material 1 Location	Material 2 Location	Material 3 Location	Material 4 Location
1 (highest)	Lab 1*	1	5	1
2	4	5	2	5
3	2	4	1	3
4	3	2	3	2
5 (lowest)	5	3	4	4

* Cell entry is the coded laboratory identifier.

TABLE 12b – Laboratory Ranking by Variation

Rank	Material 1 Variation	Material 2 Variation	Material 3 Variation	Material 4 Variation
1 (highest)	Lab 1*	5	1	1
2	4	4	3	4
3	2	1	5	3
4	5	3	2	2
5 (lowest)	3	2	4	5

* Cell entry is the coded laboratory identifier.

TABLE 12c – Material Ranking by Location and Variation

	Location		Variation	
Rank	Material	(W/m·K)	Material	(W/m·K)
1 (highest)	1	0.04151	1	0.00155
2	3	0.03626	3	0.00072
3	4	0.03378	2	0.00030
4 (lowest)	2	0.03206	4	0.00017

The results from Tables 12a and 12b succinctly demonstrate that the rankings for location and variation are not consistent across all materials (even though laboratory 1 dominates many of the #1 rankings). In other words, for a particular laboratory there is a change in location and variation from material to material. The physical reasons for this behavior are explored in detail in the next section.

The location rankings from Table 12c for materials 1 through 4 are in general agreement with current measurement results in the literature. The variation rankings contain information on between-laboratory variability and, unfortunately, are somewhat skewed because of high and low sets of data for materials 1 (laboratory 1) and 3 (laboratory 4), respectively. Elimination of this data as outlying would suggest that the between-laboratory variability for all materials would be comparable.

5.6 Comparison of Source and Recipient Laboratory Data

This section presents a direct comparison (for informational purposes only) of thermal conductivity data obtained from the source and recipient laboratories for materials 1 through 4. Figure 11a plots thermal conductivity measurements from the recipient laboratories (Table 8) versus measurements from the source laboratories (Table 7). The plot character depicts the (recipient) laboratory. The materials (1 through 4) are identified by their corresponding number. A dashed line located at 45 degrees to the horizontal and vertical axes indicates perfect agreement between the recipient and source laboratories. Values that fall below the line indicate that the recipient measurements are low with respect to the source measurements. Conversely, values above the line indicate that the recipient measurements are high with respect to the source measurements. For material 1, the recipient measurements are either spread along the line or high; for material 2, the values are slightly low; for material 3, low; and, for material 4, near or on the line. It is interesting to note that the values for material 1 are spread out along the horizontal axis in comparison to material 2, for example. Figure 11b plots the relative deviations of the recipient measurements from the 45-degree line.

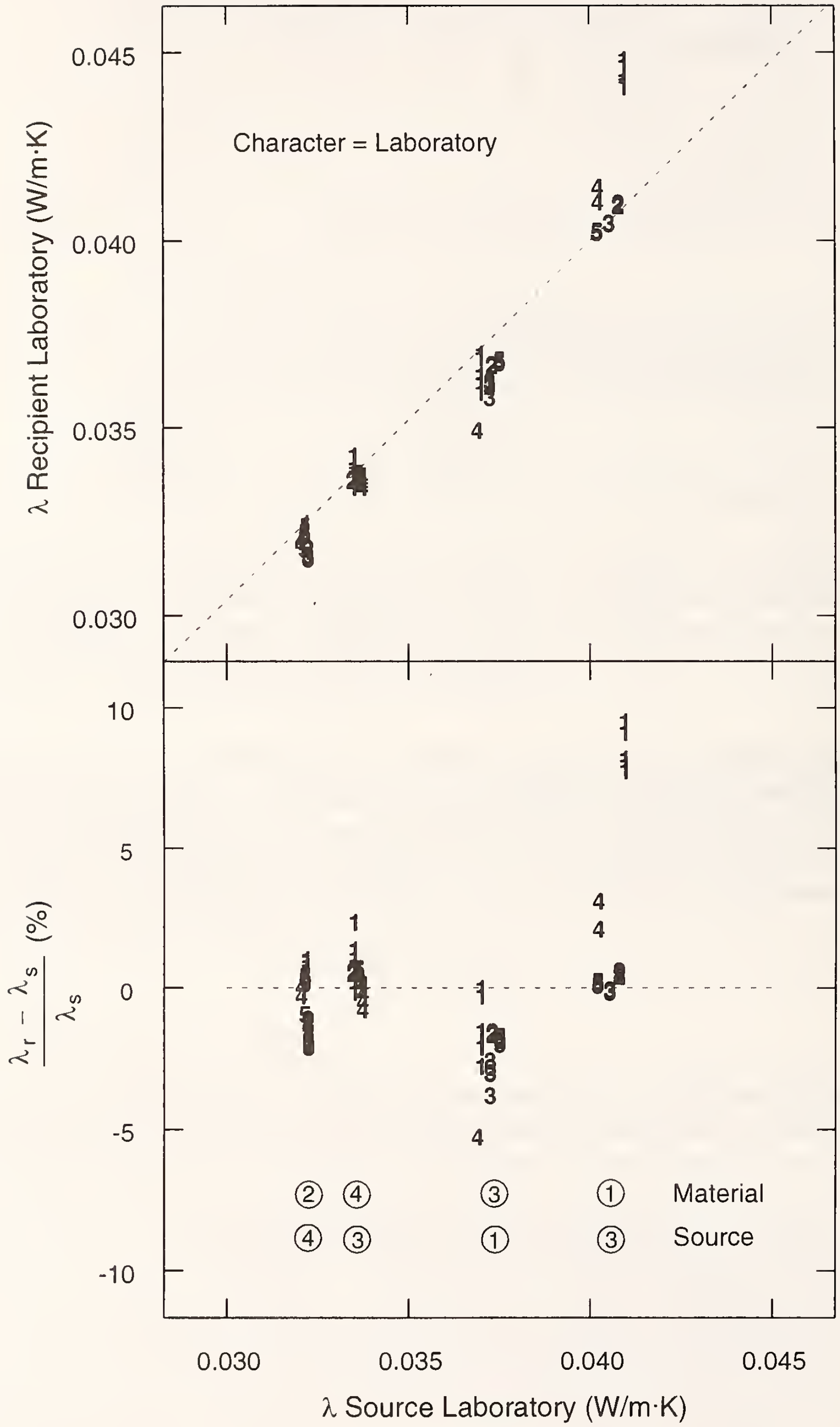


Figure 11. a) Recipient laboratories versus source laboratory (Materials 1, 2, 3, 4)
b) Relative deviations from source laboratory (Materials 1, 2, 3, 4)

6 Assessment of Fixed Temperature (297.15 K) Replicate Data

A complete and thorough assessment of the fixed temperature (297.15 K) replicate data relies on both statistical and engineering analyses of the data. A descriptive statistical analysis quantifies differences in the location and variation of the data, thereby ranking the data and identifying curious or anomalous data points. Descriptive statistics provide minimal, if any, information on why the data points behave as observed. As mentioned previously, anomalous data points were not discarded purely on the basis of outlier statistics in order to examine all the test data.

An engineering analysis, in contrast, attempts to determine the underlying cause(s) for the behavior of the test data noted in the statistical analyses. In this regard, this section conducts the following analyses:

1) Presentation of (secondary) laboratory factors;
2) Evaluation of the major sources of variation;
3) Detailed investigation of the effect of laboratory factors on λ;
4) Comparison of the laboratory test data with certified values; and,
5) Comparison of the laboratory test data with precision indices from standard test methods [2,3]

Before proceeding with the analyses, we re-present the set of primary questions identified in the statistical analysis of the data. The answers to these questions are of considerable interest and will guide the presentation of more detailed analyses. Specific questions will be answered later in the next section.

1) Q1: For 2 of the 4 materials, laboratory 1 is high, why?
2) Q2: For material 1, laboratory 5 is low, why?
3) Q3: For material 2, laboratory 3 is low, why?
4) Q4: For material 3, laboratory 4 is low, why?
5) Q5: For 3 of the 4 materials, laboratory 1 is noisy, why?
6) Q6: For material 2, laboratory 5 appears to have one outlying observation, why?

6.1 Presentation of (Secondary) Laboratory Factors

As investigated in the previous section, the primary factors of interest for the fixed temperature (297.15 K) replicate data are laboratory (5 levels) and reference material (4 levels). (The effect of temperature as a primary factor, from 280 K to 320 K, is discussed later.) Ideally, interlaboratory comparisons are designed to investigate within- and between-laboratory variability of the primary factors by minimizing the effects of other (secondary) laboratory factors. Thus, the resulting variability in the test data may be attributed to unavoidable random errors present in every experimental test method. The effects of different settings for such secondary laboratory factors will cause systematic differences in the test data. In this comparison, the effect on λ across laboratories is systematic in nature and is most likely due to differences across settings within one (or more) laboratory factor(s). It is important to identify (and subsequently estimate the effect of) the secondary laboratory factors present in this comparison.

The cause-and-effect diagram is an extremely effective tool for identifying and classifying the laboratory factors that affect a test result. Figure 12 illustrates a typical cause-and-effect diagram showing major sources of variation and underlying laboratory factors for this comparison. The major sources of variation are shown in text boxes and include 1) procedure; 2) specimen; 3) equipment; and, 4) measurement, among others. Here, procedure refers to a particular technique utilized by a laboratory. Specimen refers to the material properties affecting variability in λ. In this case, specimen only includes the effect of bulk density and excludes any affects due to the different levels of the primary factor material. Equipment covers the component differences noted in Table 4 and measurement covers all properties measured in-situ in the guarded-hot-plate apparatus (see Appendix D) for the determination of λ. Obviously, this list is not all-inclusive; the effects associated with operator and environment are not considered.

The underlying laboratory factors identified and classified in Figure 12 are summarized in Table 13 with their respective source classification. Each factor is also classified by data type, quantitative (numeric) or qualitative (categories). This distinction is important for subsequent statistical analyses. The list of factors is not intended to be all-inclusive and, in some cases, there is some redundancy among factors in different classifications (for example, note the similarity in the factors – meter plate size and meter area – classified under equipment and measurement, respectively).

TABLE 13 – Laboratory Factors Affecting the Measurement Variability of λ

	Laboratory Factors	Data Type	Source Classification
1	Steady-state conditions	Qualitative	
2	Conditioning of specimen	Quantitative	Procedure
3	Measurement technique for surface temperatures	Qualitative	
4	Bulk density (ρ)	Quantitative	Specimen
5	Plate size	Quantitative	
6	Meter plate size	Quantitative	
7	Plate emittance	Quantitative	
8	Type of heater	Qualitative	Equipment
9	Edge guarding	Qualitative	(see Table 4)
10	Temperature sensor	Qualitative	
11	Operation mode	Qualitative	
12	T_h (Hot surface)	Quantitative	
13	T_c (Cold surface)	Quantitative	
14	T_m (Mean temperature)	Quantitative	
15	ΔT (Temperature difference)	Quantitative	Measurement
16	L (In-situ specimen thickness)	Quantitative	
17	Q (Specimen heat flow)	Quantitative	
18	A (Meter Area)	Quantitative	
19	q (Specimen heat flux, Q/A)	Quantitative	

We will systematically step through each line in Table 13, performing two analyses. The first analysis evaluates the underlying factors of three major sources of variation – procedure, specimen, and measurement. The second analysis is a comprehensive statistical analysis of correlation and variance for all 19 factors given in Table 13.

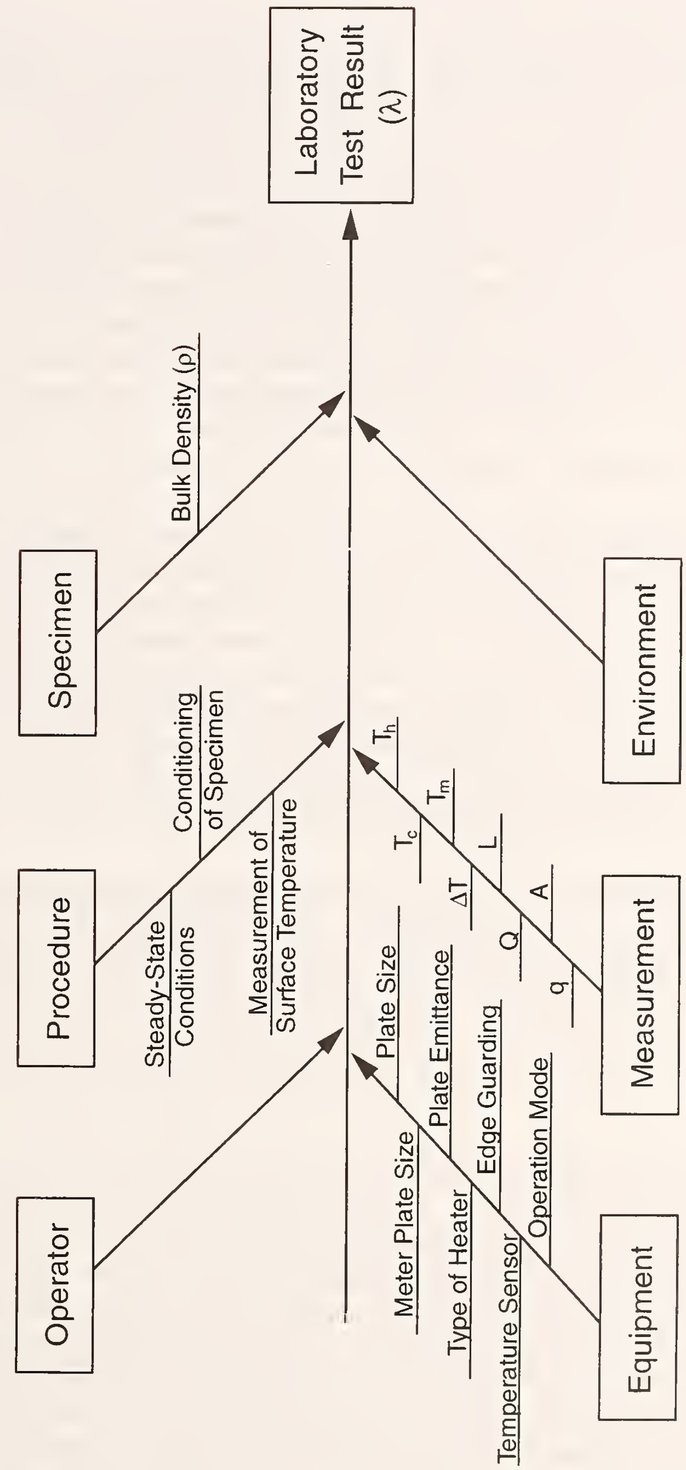


Figure 12. Cause-and-effect chart for laboratory test result (λ)

6.2 Evaluation of Major Sources of Variation

6.2.1 Procedure: Figure 12 identifies three procedural factors that may have caused variations in the test results: 1) differences in defining steady-state conditions; 2) specimen conditioning; and, 3) the technique for measuring the temperature difference across the specimen.

6.2.1.1 Steady-State Conditions: A discussion of the laboratory equipment revealed that each laboratory has developed similar, but not identical, in-house procedures for obtaining steady-state conditions (i.e., short-term control stability) of their respective apparatus. The procedures are based on typical guidelines for settling time and measurement interval established in ISO 8302 [2] and ASTM C 177 [3] test methods. Since the procedures involve different groups of parameters and stabilization times, a complete laboratory-to-laboratory comparison is not possible. In general, however, the procedural differences reported among the laboratories for obtaining steady state are small and, therefore, not believed to be a significant source of the systematic differences observed in the data.

6.2.1.2 Specimen Conditioning: The laboratory environments for specimen conditioning were unspecified in the test protocol (Appendix B) and left to the individual laboratories. Although not requested, laboratory 2 included their specimen conditioning data with their original report forms. Similar data were requested from the other laboratories after completion of this study. Table 14 summarizes the available. data. Although the data are too sparse for definitive conclusions, in general, ambient conditioning of these materials plays little or no role. The fibrous material does not pick up moisture sufficient to have an effect and the presence of a binder allows only a small amount (0.3 % to 0.4 % by mass fraction). Likewise, polystyrene (closed-cell) foam does not pick up moisture under most laboratory ambient conditions.

TABLE 14 – Conditioning Environments for Specimens (Post-Comparison, except Lab 2)

Lab	Material 1		Material 2		Material 3		Material 4	
	T (°C)	RH (%)	T (°C)	RH (%)	T (°C)	RH (%)	T (°C)	RH (%)
1								
2[1]	23	50	23	50	23	50	23	50
3	23	20	24	30	23	30	24	25
4								
5								

[1]Laboratory 2 dried specimens of materials 1, 2, and 3 in an oven at 70 °C prior to conditioning

6.2.1.3 Surface Temperature Measurement Technique: With respect to surface temperature, two different techniques were utilized in this comparison for the surface temperature measurement: 1) permanent sensors affixed to the apparatus; and, 2) removable sensors affixed to the specimen. Both ISO 8302 [2] and ASTM C 177 [3] provide recommended techniques to determine the temperature difference across a specimen. For non-rigid specimens, the temperature difference is usually determined using temperature sensors permanently mounted in the heating and cooling surfaces[2]. For rigid specimens, the temperature difference may be determined by mounting sensors flush (typically by cutting grooves) in the specimen surfaces.

[2]Temperature sensors such as thermocouples are typically installed in grooves cut in the surfaces of the plates. For laboratory 3, a platinum resistance thermometer is actually installed in the guard-gap on the perimeter of the meter plate in accordance with ASTM practice C 1043 [14].

Four of the five laboratories utilized the first technique, that is, temperature sensors permanently mounted in the heating and cooling surfaces. After submission of their test data, laboratory 1 reported that the surface temperatures of their specimens were measured using 0.2 mm diameter thermocouples (Type T) placed directly on the surface of the specimen with adhesive tape. For the replicate measurements, the thermocouples were removed and reapplied prior to each measurement. It is surmised that much of the variability observed in Figure 4 could be attributed to this measurement technique of affixing thermocouples to the surface of the specimen. In an early comparison of guarded hot plates, Robinson and Watson [15] noted that discrepancies could result between conductivity values obtained using temperatures from plate surfaces and those measured using surface thermocouples. Intuitively speaking, the surface mounted technique would seem to have inherently more variation than sensors permanently affixed to the apparatus and, thus, contribute to less precise levels of replication.

This premise is investigated by plotting the laboratory test results for λ on a relative basis (%) versus material as illustrated in Figure 13a. The five laboratory replicates are plotted sequentially for each material. Note that for laboratory 1, the relative differences from the grand mean are substantially different across the four materials (Figure 13b) and the relative standard deviations are high for materials 1, 3, and 4 (Figure 13c). As noted before, materials 1, 2, and 3 are fibrous materials having average bulk densities of 14 kg/m^3, 72 kg/m^3, and 227 kg/m^3, respectively (Figure 1). Material 1 is a flexible blanket and materials 2 and 3 are semi-rigid boards of increasing rigidity. By contrast, material 4 is expanded polystyrene in the form of a semi-rigid board having relatively smooth surfaces in comparison to the fibrous materials. The results in Figure 13 would tentatively suggest an interaction between the surface measurement technique of affixing the sensors to the materials and the type of material.

The approach of adhering fine-diameter temperature sensors to the specimen surface appears to have contributed to measurement differences and may be an *unintended* extension of the test procedures specified in the ISO and ASTM standard test methods [2,3]. Further measurements comparing different techniques for determining the temperature difference across a test specimen would be extremely useful. With regard to ISO 8302 [2] and ASTM C 177 [3] standard test methods, the appropriate sections on determination of the temperature difference should be re-examined for clarity and revised if necessary.

6.2.2 Specimen: The inclusion of bulk density (ρ) with the other factors in Table 13 is necessary, even though a substantial effort was undertaken by the source laboratories to issue well-matched specimens of a homogeneous sample of each reference material (Figures 1 and 3, respectively). Many of the specimens, however, were subsequently cut smaller for the different plate sizes (Table 4) involved in this comparison. Within-specimen variability, if present, could contribute to differences in density between the source and recipient laboratories. Furthermore, variability between specimen and meter area bulk densities may also be present. As mentioned earlier, there was no provision included in the protocol (Appendix B) for determining the bulk density of the meter area of the specimen. Since the meter area bulk density is more representative of the measured thermal conductivity, future comparisons should, if possible, request the determination of the meter area bulk density for the analysis of the λ-versus-ρ data. For the samples of reference materials released to the participants, the pertinent question becomes: Is there a specimen bulk density effect within a material for the fixed temperature (297.15 K) replicate data?

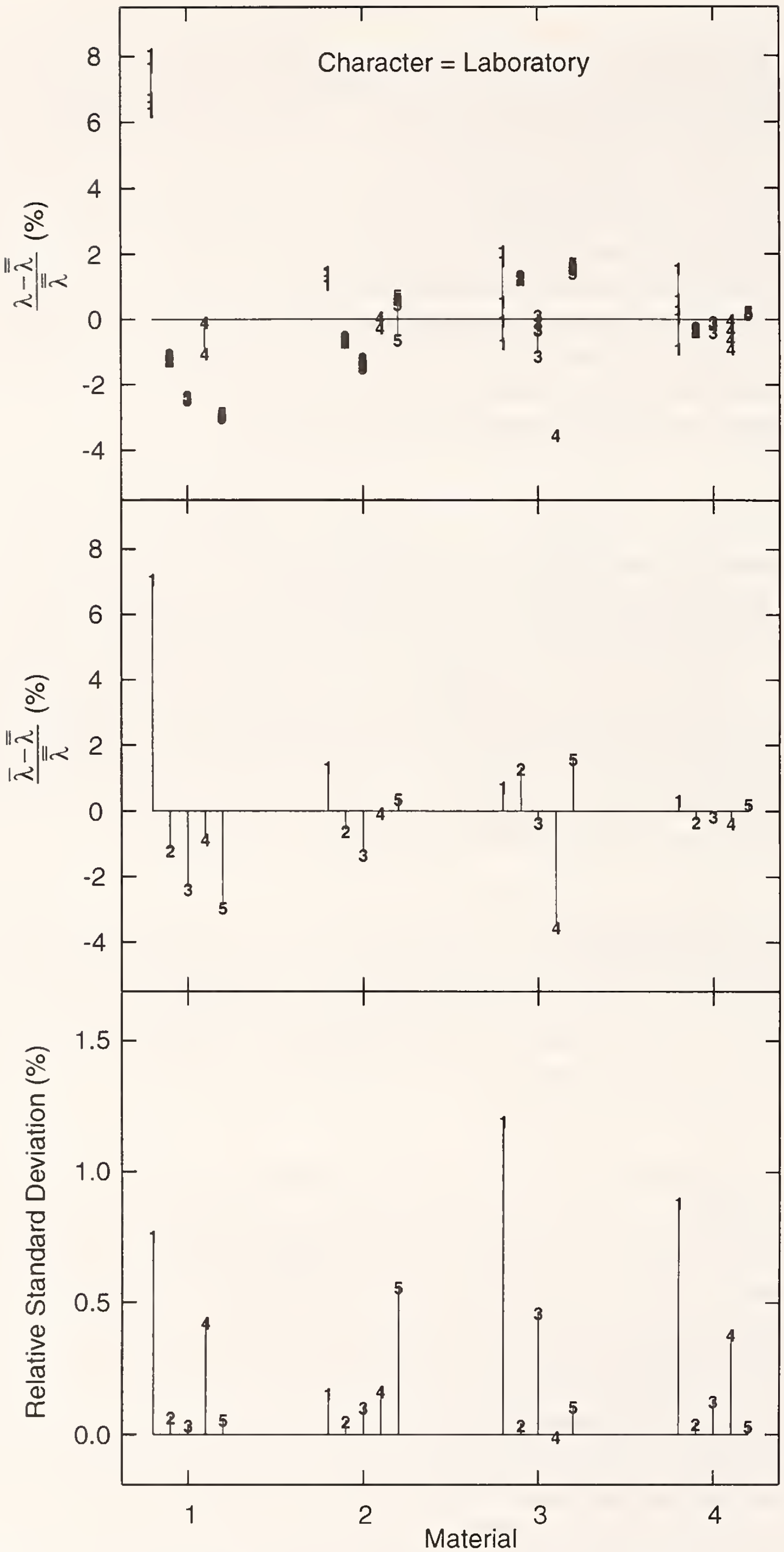


Figure 13. a) Relative thermal conductivity versus material;
b) Relative mean thermal conductivity versus material;
c) Relative standard deviation thermal conductivity versus material

Figure 14 plots λ versus specimen ρ for each material. Vertical dashed lines denote the initial density range measured by the source laboratory (Table 5). For materials 1 and 4, predicted values of $\lambda(T_m,\rho)$ at 297.15 K from Eq 1 are shown as solid lines. The predicted values for material 1 increased from 0.04039 W/m K to 0.04138 W/m K (about 2.5 %) over the range of ρ from 13.4 kg/m^3 to 14.4 kg/m^3. For material 4, the change in $\lambda(T_m,\rho)$ was less than 0.1 %. Unfortunately, a definitive analysis of Figure 14 is problematic for the following reasons.

1) Figure 14 indicates a negative λ-ρ relationship for material 1, positive for material 2, and weak for material 3. In contrast, Figure 3 (source lab data) indicates the λ-ρ relationship was weak for materials 1 and 2; and, positive for material 3. These differences suggest that either within-specimen variability is significant (particularly for material 1), or the results of Figure 14 are perhaps skewed by the densities reported by laboratory 1, or both.

2) The bulk densities reported by laboratory 1 are either consistently a minimum or maximum value across all four materials, why? A partial explanation is available from the results given in Figure 1 for materials 2 and 3. Laboratory 1 received specimen pairs for materials 2 and 3 having the maximum and minimum density, respectively, for each material (Figure 1).

3) The bulk densities reported by laboratory 1 are, in some cases, considerably outside the ranges provided by the source laboratories in Figure 1. Were there problems with the density measurement, part from the metering section issue? Further information on this issue, and the above, from laboratory 1 is on request.

Although unrelated to density, there are other noteworthy issues concerning some of the specimens, which are discussed here. The comments below were reported by laboratory 4.

1) For material 3, laboratory 4 reported values of λ that are 3.5 % below the grand mean for material 3 (Table 11.) In the comment section of their official test report form, laboratory 4 reported that, "this material had completely delaminated on arrival so that the test specimen consisted of two pieces which were always aligned in the same orientation with respect to each other whilst testing." Since no other laboratories reported similar experiences, this set of data for material 3 is considered sufficiently different from the other specimens to warrant rejection as an outlying observation.

2) For material 4, laboratory 4 reported values of λ that are relatively imprecise when compared to their own data for the other 3 materials. In the comments section of their official test report form, laboratory 4 reported that the higher variability in the data "could (be) due to an increased uncertainty associated with the measurement thickness of about 13 mm." The laboratory noted that "this sample is thinner than the limit of 25 mm set for minimum specimen thickness in accordance with ISO 8302 [2]." This condition is simply noted since this data is only affected marginally.

Finally, some final comments on material types and density levels are in order. Although this comparison studied four reference materials, there were only two types of materials: fibrous glass (ρ = 3 levels) and cellular expanded polystyrene (ρ = 1 level). In hindsight, future comparisons may alternatively consider balancing the number of levels of ρ for each material type

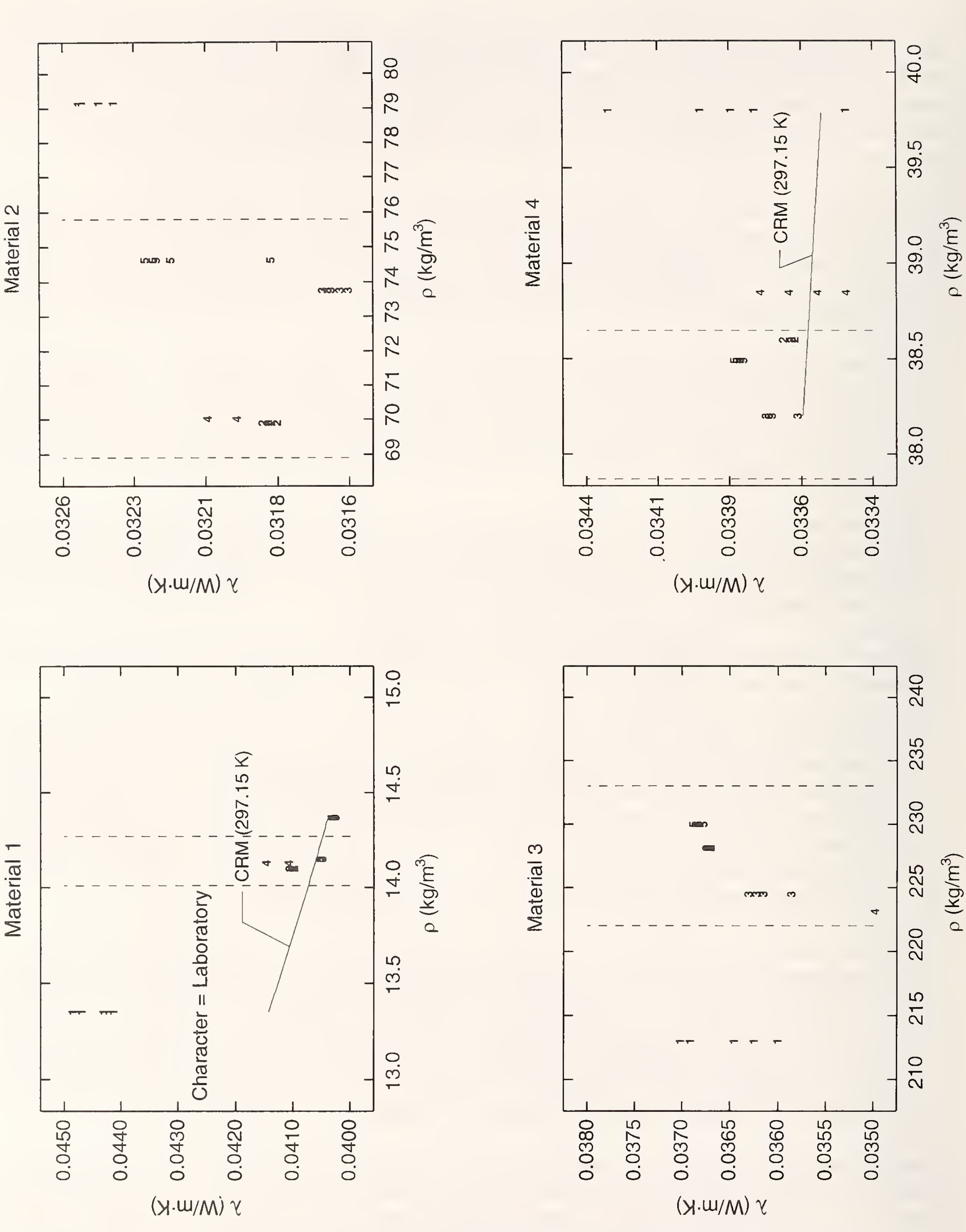


Figure 14. Thermal conductivity (297.15 K) as a function of bulk density (Materials 1, 2, 3, 4)

or, at a minimum, consider 2 levels of ρ per material. In addition, if possible, it would probably be useful to characterize the microstructure (pore size, fiber diameter, etc.) of each material and determine the extent of within-specimen variations of bulk density.

6.2.3 Equipment: Figure 12 identifies several equipment factors (from Table 4) that may have caused variation in the test result. The effects of these factors are investigated later as part of the comprehensive analysis of all the laboratory factors.

6.2.4 Measurement: This section investigates six of the eight factors identified in Figure 12: T_h, T_c, T_m, ΔT, L (in-situ), and q. Values for these parameters were requested as part of the official returns from the laboratories (Appendix C) and tabulated in Table 8. The analysis is carried out in two parts:

1) A check of the protocol execution given in Appendix B; and,
2) An in-depth analysis of T_m, ΔT, and L (in-situ).

6.2.4.1 Check of Protocol Execution: Figure 15 is a sequence of plots for λ versus the thermal test parameters; T_h, T_c, T_m, ΔT, L, and, q. The first four plots (Figures 15a to 15d) check how effectively the laboratories executed the test protocol (Appendix B) to obtain target temperatures of T_m = 297.15 K (24 °C) and ΔT = 20 K (regardless of material). Dashed vertical lines indicate target temperatures in the first four plots. For example, Figure 15a (T_h) indicates a target temperature of 307.15 K (34 °C).

The data indicate that laboratory 1 was low with respect to both T_h and T_c, and that laboratory 4 was low for T_h but high for T_c. The net effect, as shown in Figures 15c and 15d, was that laboratory 1 was significantly low for T_m and high and low for ΔT. Laboratory 4 was slightly low for T_m (<0.5 K) and low for ΔT. The other laboratories were generally tightly clustered about the respective target temperatures. Table 15 summarizes the laboratory statistics for the mean, standard deviation, and range for values of T_h, T_c,, T_m and ΔT given in Figures 15a to 15d.

TABLE 15a – Mean, Standard Deviation, and Range of T_h and T_c (Table 8)

Lab	$\overline{T}_h$	$\overline{T}_h - 307.15$	SD(T_h)	Range	$\overline{T}_c$	$\overline{T}_c - 287.15$	SD(T_c)	Range
	(K)	(K)	(K)	(K)	(K)	(K)	(K)	(K)
1	306.64	−0.51	1.23	4.32	286.24	−0.91	0.39	1.55
2	307.18	0.03	0.03	0.12	287.20	0.05	0.07	0.25
3	307.15	0.00	0.00	0.00	287.15	0.00	0.00	0.01
4	306.17	−0.98	0.33	0.99	287.56	0.41	0.22	0.61
5	307.14	−0.01	0.03	0.13	287.15	0.00	0.01	0.04

TABLE 15b – Mean, Standard Deviation, and Range of T_m and ΔT (Table 8)

Lab	$\overline{T}_m$	$\overline{T}_m - 297.15$	SD(T_m)	Range	$\Delta\overline{T}$	$\Delta\overline{T} - 20$	SD(ΔT)	Range
	(K)	(K)	(K)	(K)	(K)	(K)	(K)	(K)
1	296.44	−0.71	0.64	2.09	20.39	0.39	1.32	4.65
2	297.19	0.04	0.04	0.15	19.98	−0.02	0.06	0.23
3	297.15	0.00	0.00	0.00	20.00	0.00	0.00	0.01
4	296.86	−0.29	0.14	0.43	18.61	−1.39	0.49	1.31
5	297.14	−0.01	0.02	0.07	19.99	−0.01	0.03	0.13

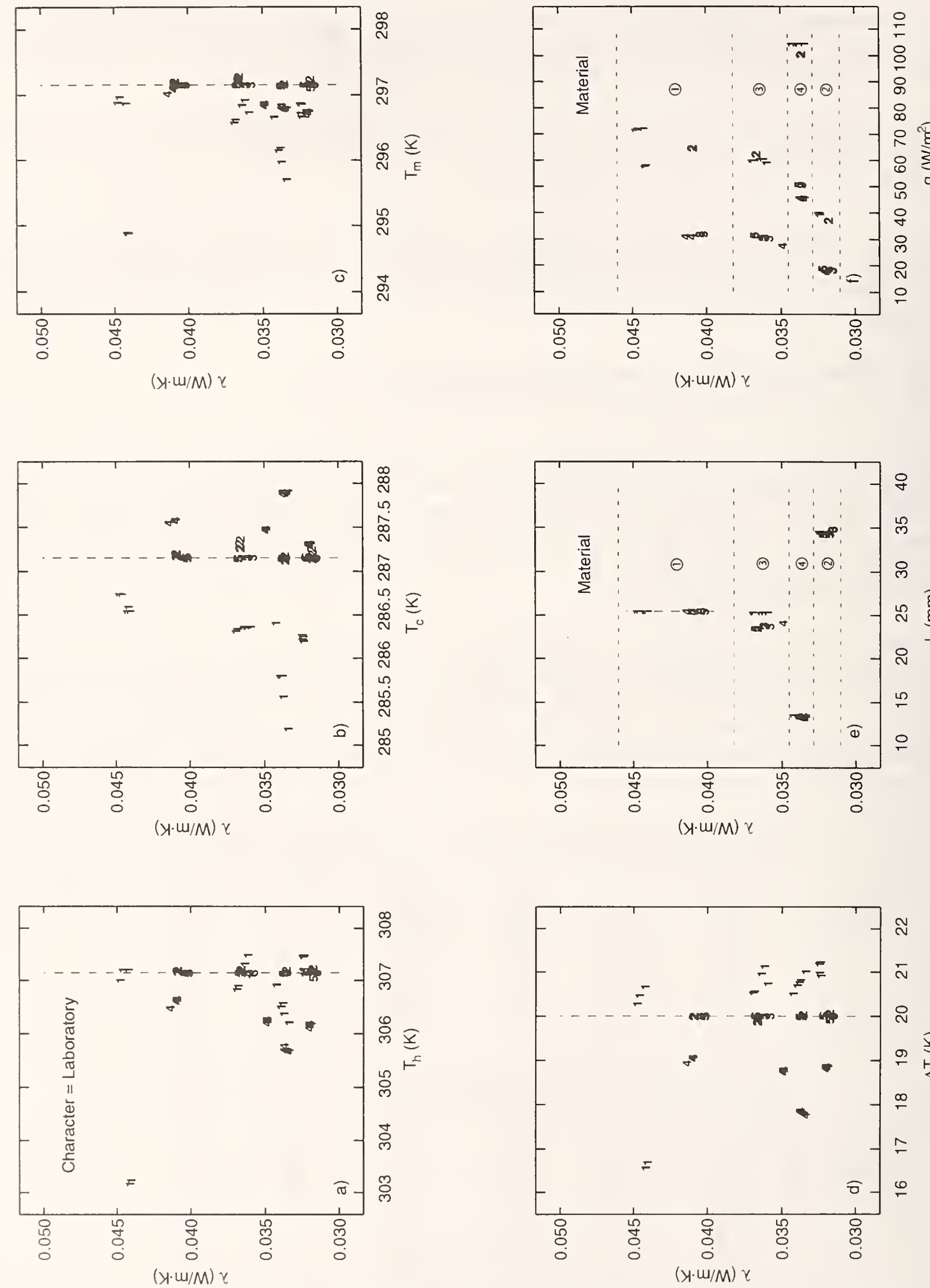


Figure 15. Multi-plot of thermal conductivity versus test parameters (Materials 1, 2, 3, 4)

Figure 15e plots λ versus L for all the laboratories. Horizontal lines have been inserted that delineate the thermal conductivity (λ) ranges of the four materials (shown as ①,②,③, and ④, respectively). (Note that these lines are not shown in Figures 15a to 15d, although the same ranges do apply.) A target thickness of 25.4 mm was specified only for material 1 (Appendix B). The thicknesses for the other materials were unspecified in the test protocol and measured by the laboratories as received. These thickness measurements are in close agreement except for the few cases that are discussed below.

Figure 15f plots λ versus q for all the laboratories. Horizontal lines have been inserted that delineate the thermal conductivity (λ) ranges of the four materials (shown as ①,②,③, and ④, respectively). It is interesting to note that laboratories 1 and 2 reported values of q that were essentially twice the values determined by the other laboratories. These differences are merely a discrepancy in the accounting for the factor of 2 given in Eq 2, and indicate that further clarity for the definition of q is needed in either the test protocol (Appendix B), or the standard test methods [2,3], or both.

6.2.4.2 Mean Temperature (T_m): Figure 16 plots λ as a function of T_m for each material. For the fixed-temperature replicate data, all laboratories were requested to test the specimens at a mean temperature of 297.15 K (24 °C). Examination of the plots reveals that the data, for most laboratories, are near 297.15 K (24 °C). As discussed previously (Figure 15), laboratories 1 and 4 reported mean temperatures that are consistently lower than the other laboratories. There are also other interesting trends in the data. For material 2, the direction of variation for laboratories 3 and 5 is vertical and for laboratory 2, horizontal. In some cases, these variations change direction from material to material (for example, note directional change in variability for laboratories 2 and 4 for materials 3 and 4). The reason(s) for these trends is not known.

6.2.4.3 Temperature Difference (ΔT): Figure 17 plots λ versus ΔT for each material. All laboratories were requested to test their specimens at a temperature difference of 20 K, regardless of material (and mean temperature). Examination of the plots reveals that the data, for most laboratories, are near 20 K. As discussed previously (Figure 15), laboratories 1 and 4 reported temperature differences that were consistently higher (about 1 K) and lower (about 1 K to 2 K), respectively, than the other laboratories. For material 1, laboratory 1 reported 2 observations that were about 3 K low. In some cases, as was observed in Figure 16, variations in direction changed from material to material (for example, note directional change in variability for laboratory 4 from materials 3 to 4). The reason(s) for these trends is not known.

6.2.4.4 Thickness (L): Figure 18 plots λ versus L for each material. With the exception of material 1, all laboratories were requested to measure the specimen thickness as received. A target thickness of 25.4 mm was specified for material 1 (Appendix B). All laboratories reported such thicknesses to 2 decimal places – with the exception of laboratory 1 which reported 1 decimal place (Table 8). For laboratory 1, there is no variation in any of the measurements of thickness (perhaps due to the rounding). The thickness data for laboratory 2 is generally quite tight for the fibrous glass materials. The same statement applies for laboratory 5, except for material 1. Both laboratories 2 and 5, however, report more variation in L for material 4 (cellular expanded polystyrene). For material 1, laboratory 3 is about 0.4 % higher than the other laboratories. Requesting additional information on in-situ thickness measurement techniques would be useful for future interlaboratory comparisons.

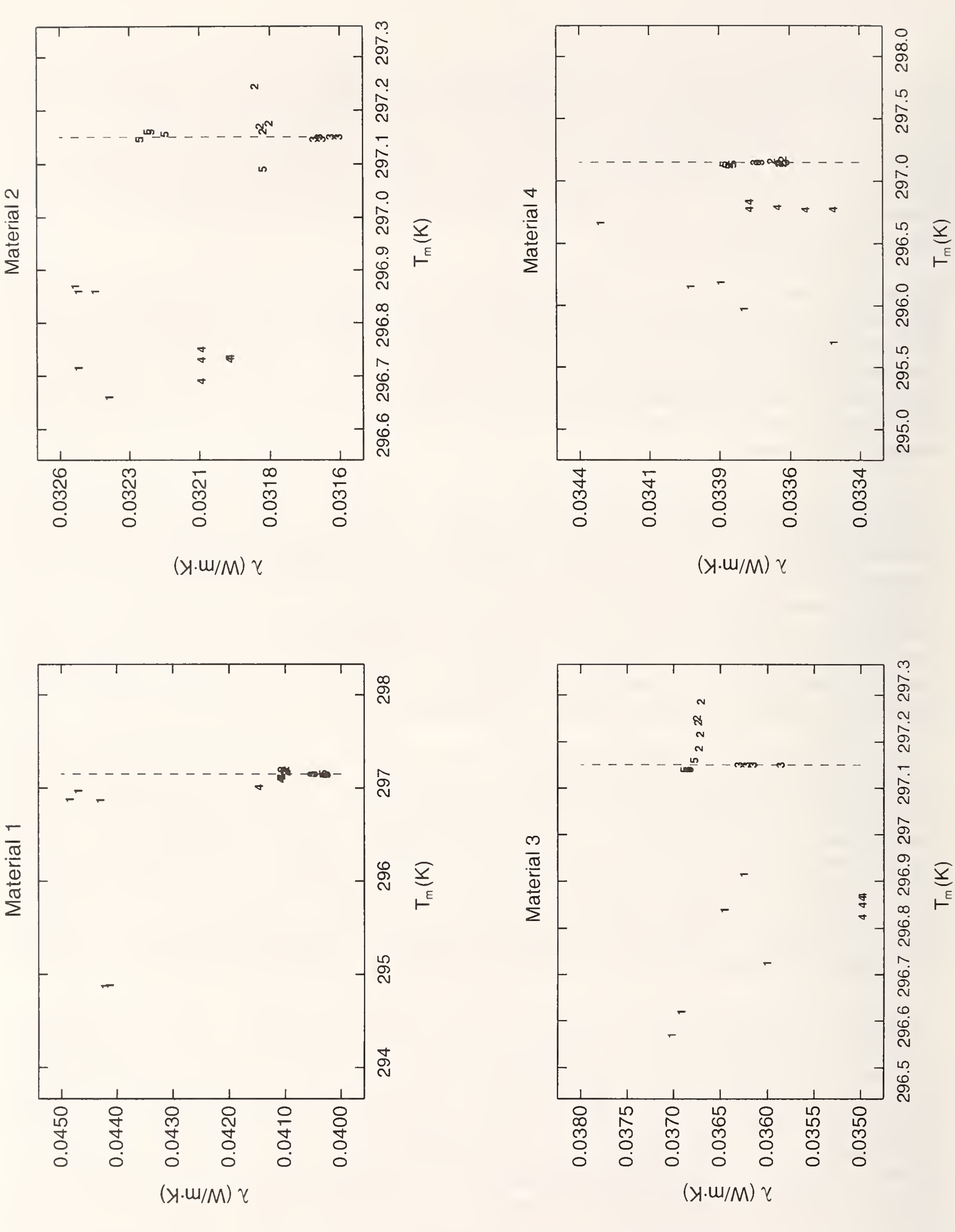


Figure 16. Replicate data (297.15 K) as a function of mean temperature (Materials 1, 2, 3, 4)

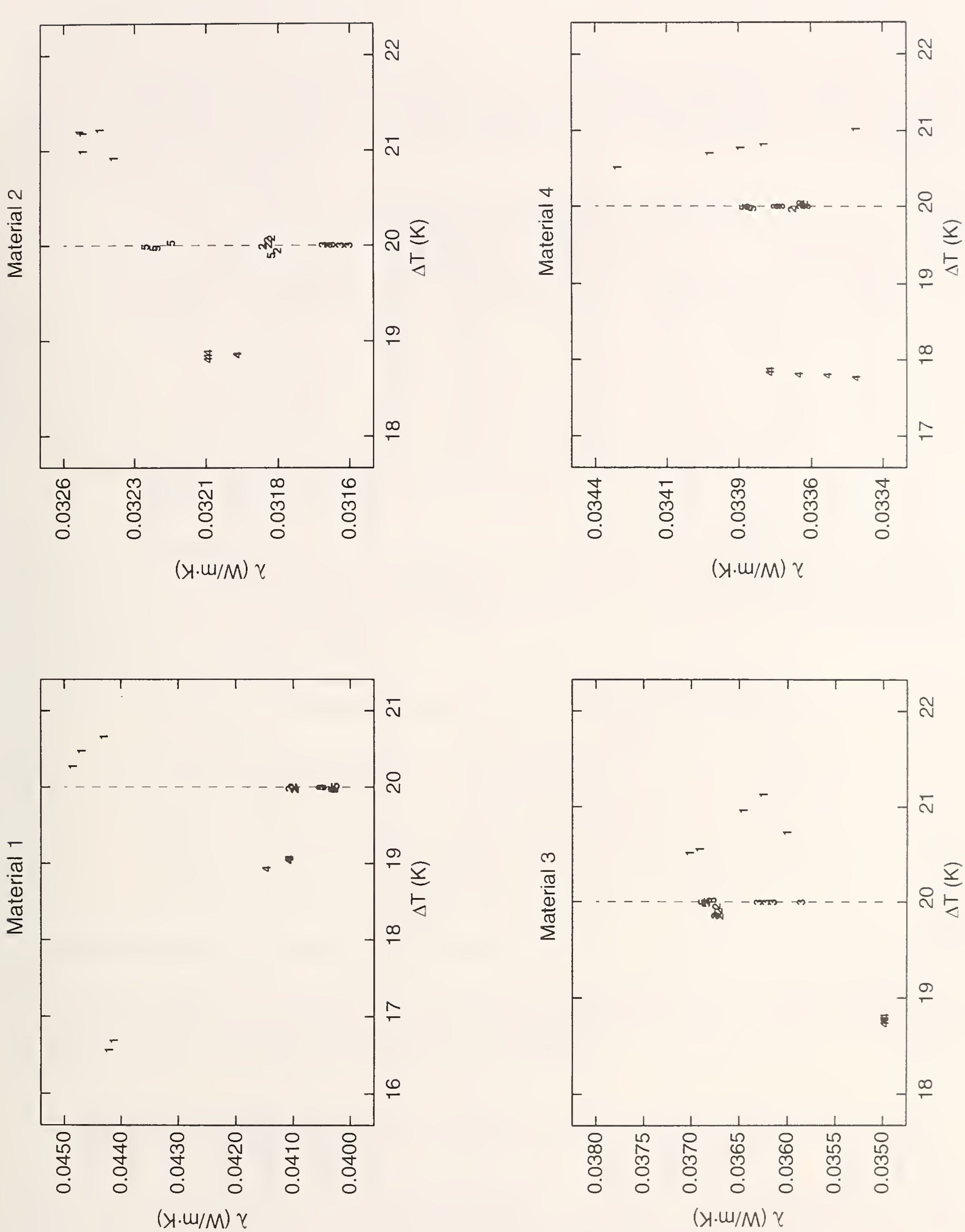


Figure 17. Replicate data (297.15 K) versus temperature difference (Materials 1, 2, 3, 4)

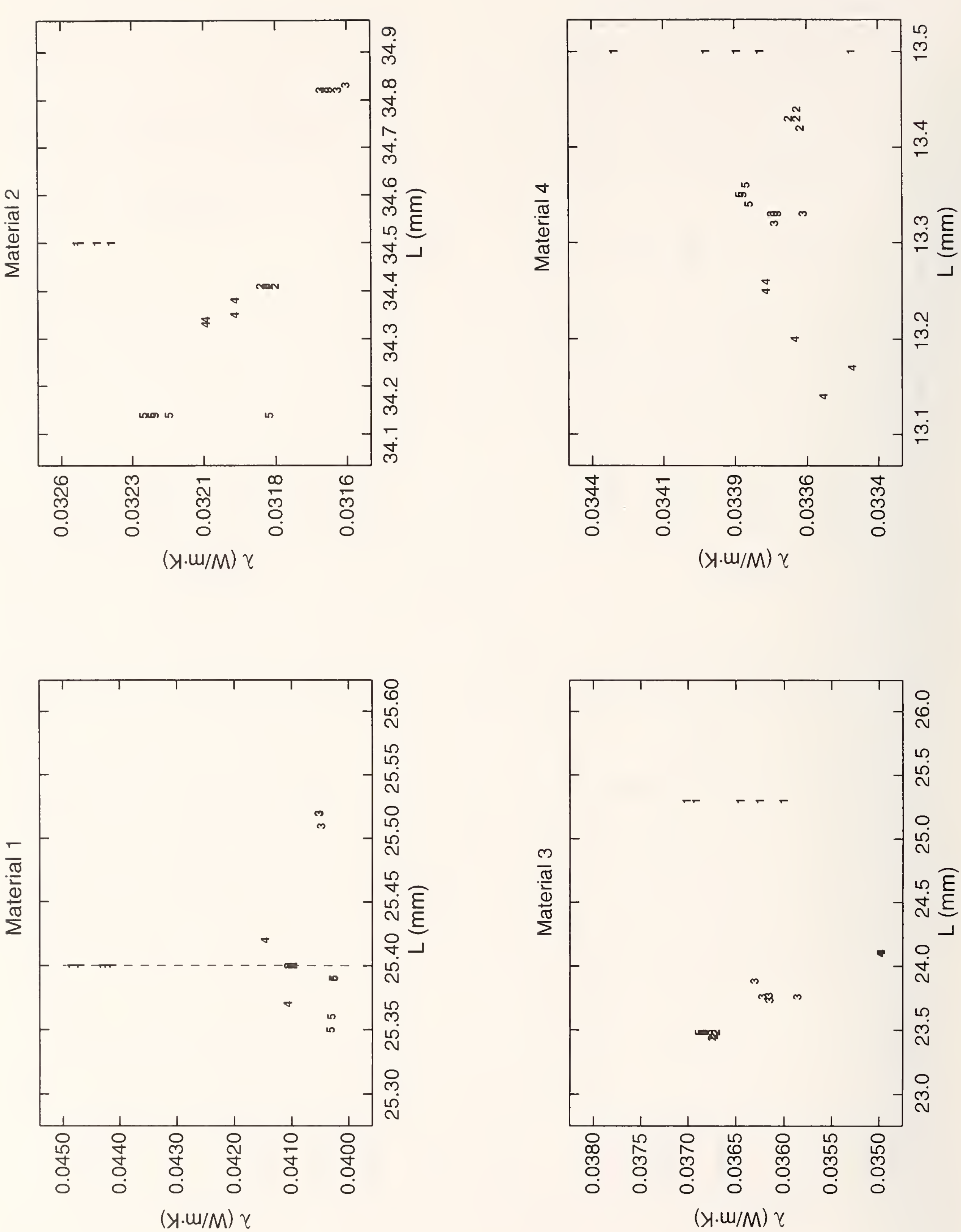


Figure 18. Replicate data (297.15 K) versus thickness (Materials 1, 2, 3, 4)

Table 16 summarizes the laboratory statistics for the mean and standard deviation of L by material. Superscripts (^{H}) and (^{L}) designate the high and low laboratory means, respectively, for each material. The grand mean and standard deviation (n = 25 observations per material) are given in the last row of Table 16.

TABLE 16 – Mean and Standard Deviation of L (Table 8)

Lab	Material 1		Material 2		Material 3		Material 4	
	$\bar{L}$ (mm)	SD(L) (mm)	$\bar{L}$ (mm)	SD(L) (mm)	$\bar{L}$ (mm)	SD(L) (mm)	$\bar{L}$ (mm)	SD(L) (mm)
1	25.4	0.0	34.5	0.0	25.3^H	0.0	13.5^H	0.0
2	25.40	0.00	34.41	0.00	23.46^L	0.02	13.43	0.01
3	25.52^H	0.00	34.82^H	0.00	23.78	0.06	13.33	0.00
4	25.38^L	0.02	34.35	0.02	24.10	0.01	13.20^L	0.05
5	25.38^L	0.02	34.14^L	0.00	23.48	0.00	13.35	0.01
Grand	25.41	0.06	34.44	0.25	24.02	0.76	13.36	0.11

^{H}High value for material; ^{L}Low value for material

With the exception of material 3, the values for the grand standard deviations in Table 16 are relatively small. On a relative basis (dividing the grand standard deviations by their respective grand mean), the relative standard deviations are 0.2 %, 0.7 %, 3.2 %, and 0.8 % for materials 1, 2, 3, and 4, respectively. For comparison, Table 17 provides summary thickness statistics for the source laboratory data given in Table 7.

TABLE 17 – Mean and Standard Deviation of Source Laboratory L (Table 7)

Material	Source Lab	$\bar{L}$ (mm)	SD(L) (mm)	Rel. SD(L) (%)
1	3	25.53	0.01	0.04
2	4	34.42	0.07	0.2
3	1	23.8	0.3	1.4
4	3	13.43	0.06	0.4

6.3 Detailed Investigation of Laboratory Factors

In contrast to the previous evaluation and discussion of major sources of variation identified in the cause-and-effect chart (Figure 12), this comprehensive statistical analysis of the 19 laboratory factors (from Table 13) addresses two questions:

1) Is there a factor effect on λ? and,
2) What is the relationship between the response variable (λ) and a factor?

Results of the analysis are presented by graphical analysis of the data as illustrated in Figures 19, 19, 20, and 21 for materials 1, 2, 3, and 4, respectively. Each figure contains 20 scatter plots of λ (vertical axis) versus the factors 1 through 19 (horizontal axis) as given in Table 13. As a point of reference, the first plot of each figure graphs λ versus the primary factor laboratory.

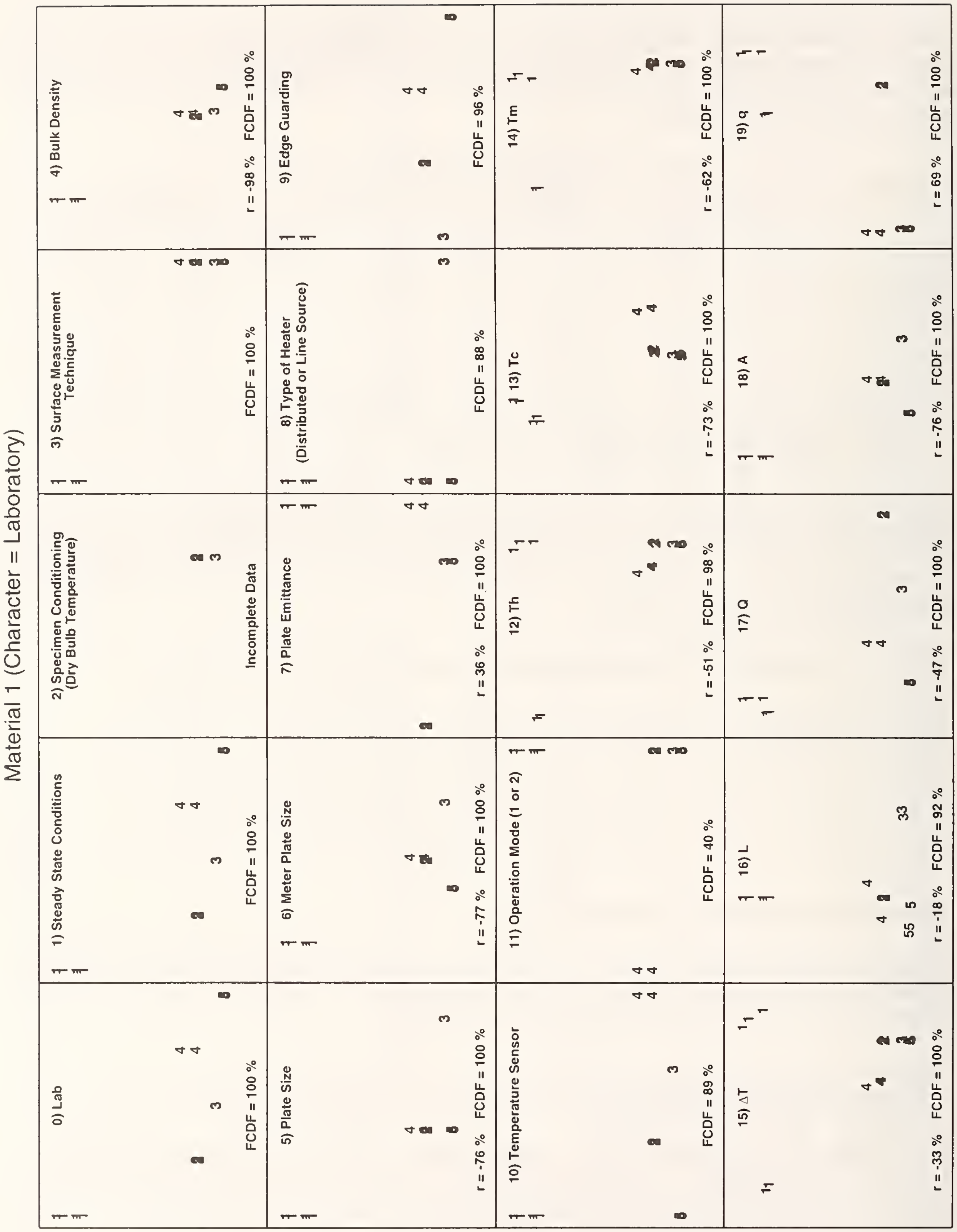


Figure 19. Scatter plots of λ (297.15 K) versus laboratory factors - Material 1

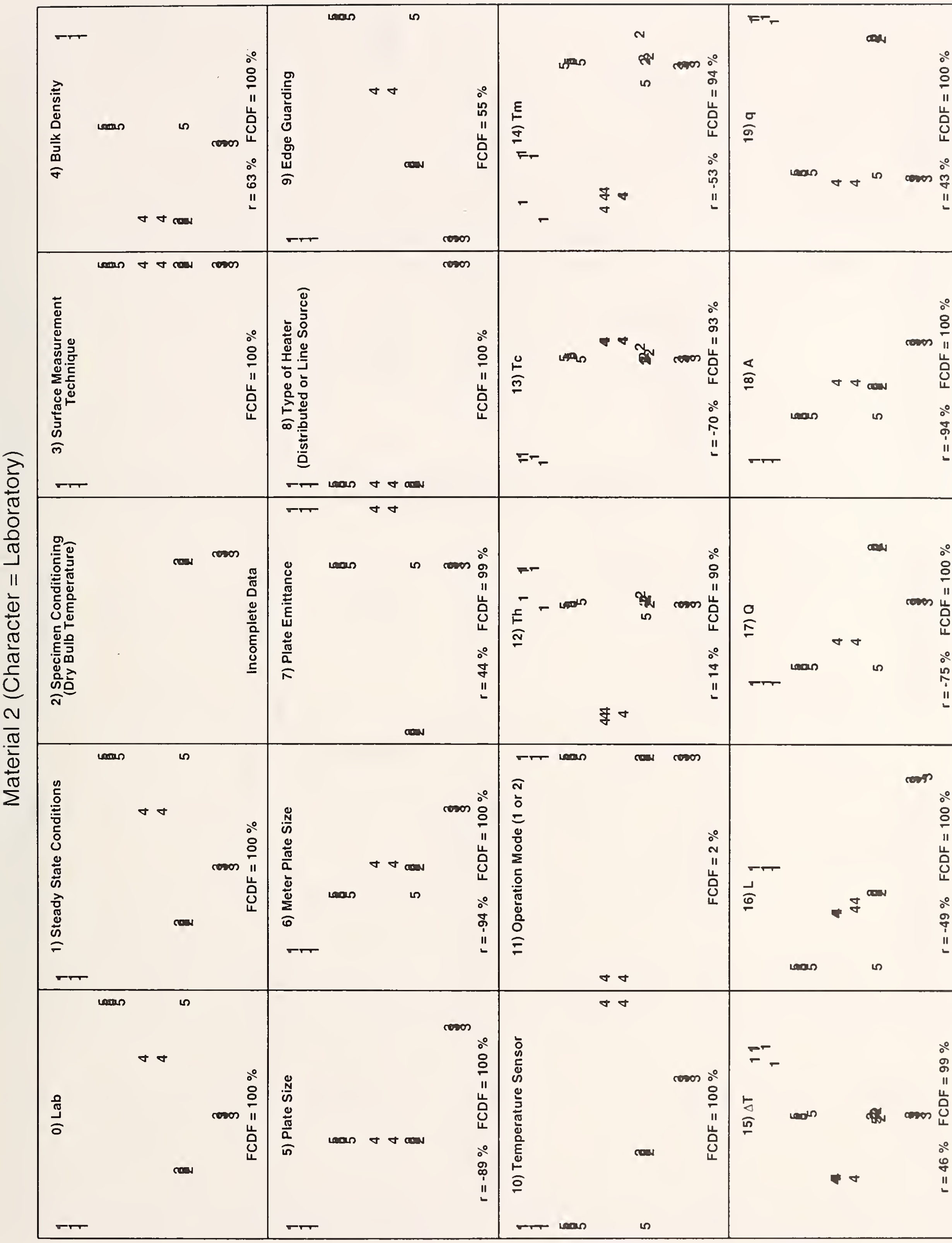


Figure 20. Scatter plots of λ (297.15 K) versus laboratory factors - Material 2

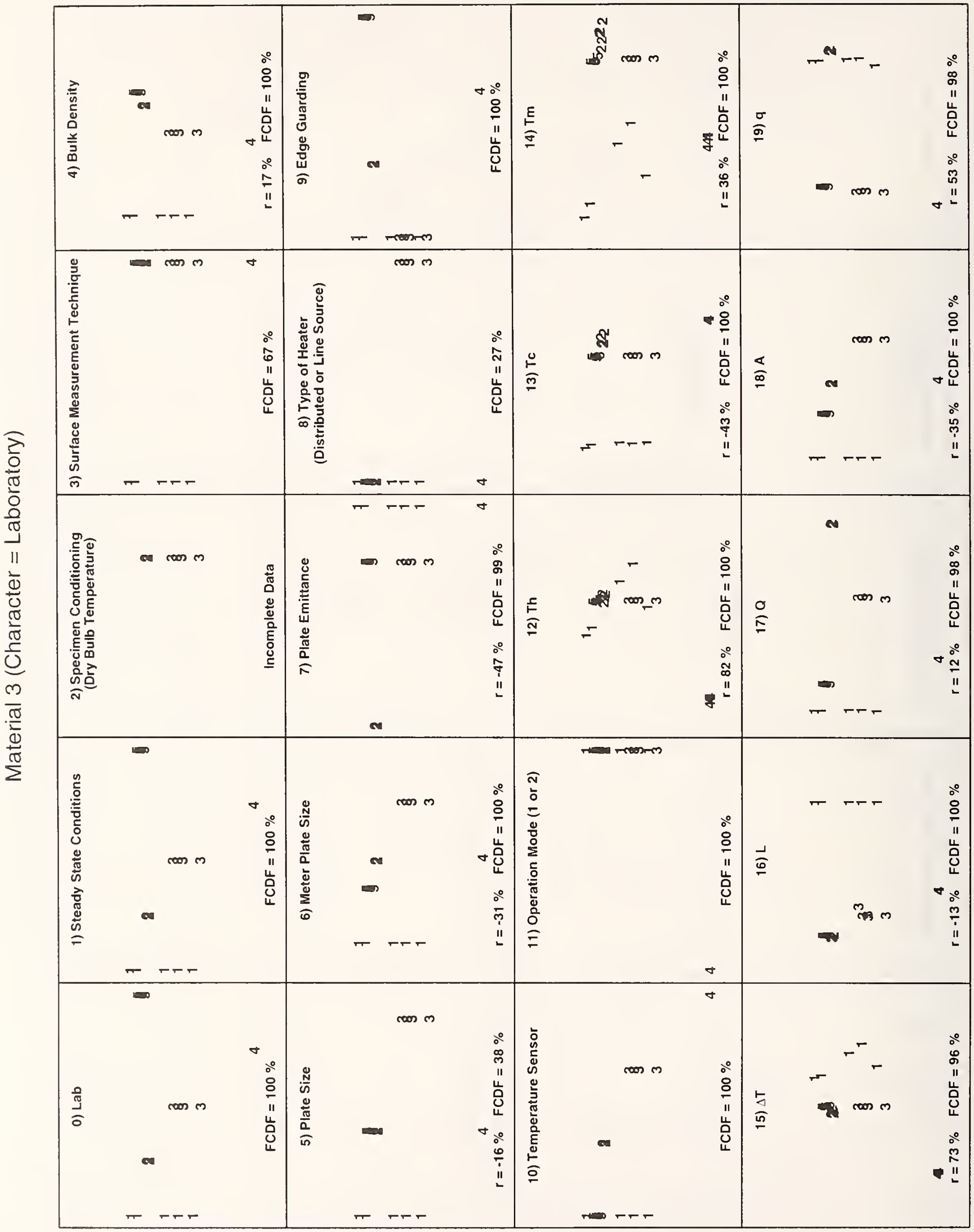


Figure 21. Scatter plots of λ (297.15 K) versus laboratory factors - Material 3

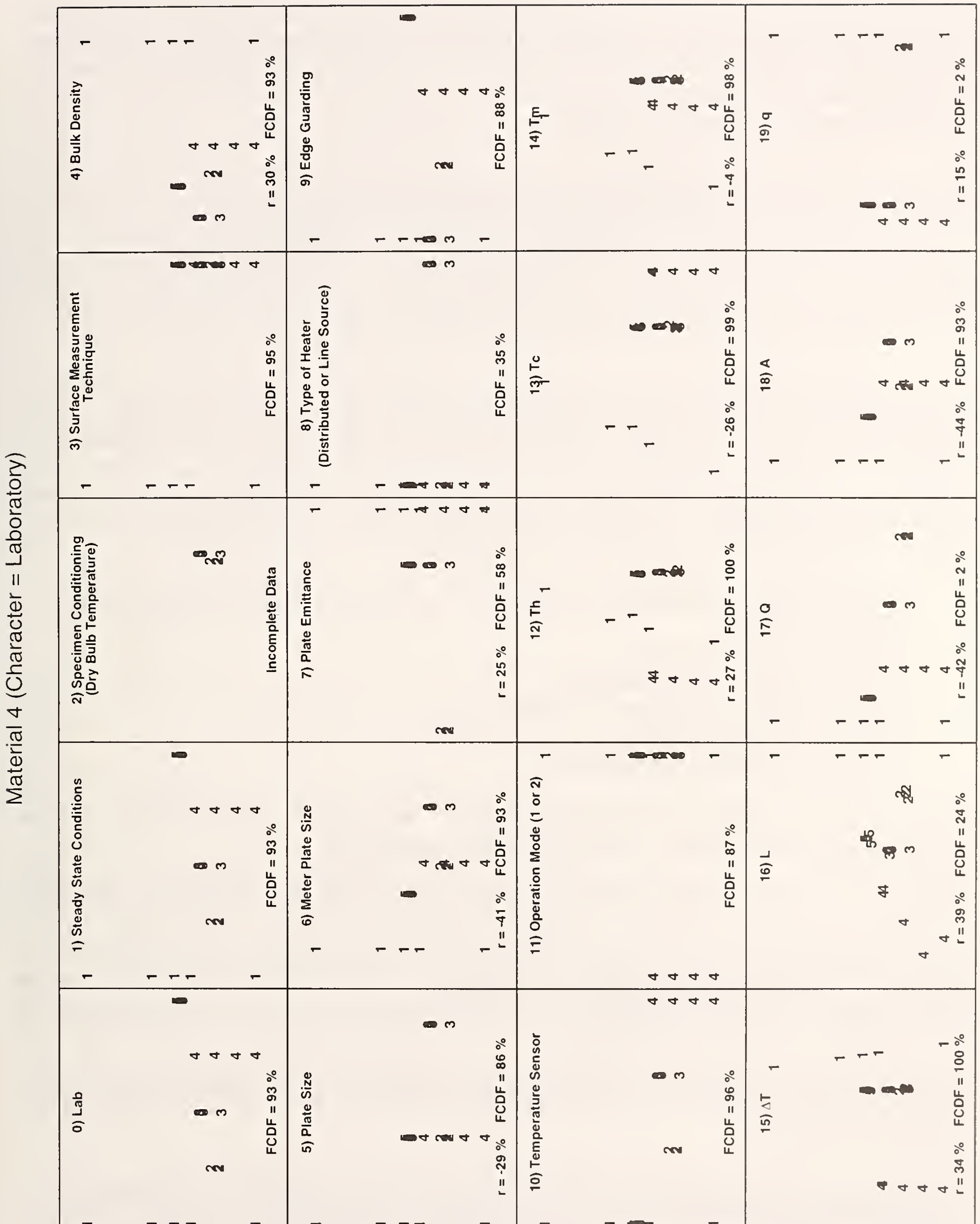


Figure 22. Scatter plots of λ (297.15 K) versus laboratory factors - Material 4

For a given material, each plot provides a correlation coefficient (r) (for quantitative x-axis data only) and an analysis of variance (ANOVA) cumulative probability for λ and the laboratory factor of interest. Values of r near $\pm$ 100 % and values of the ANOVA cumulative probability near 100 % both are indications of factor significance. The following information is provided to assist in interpreting the graphical analysis.

The plot character in all plots is laboratory (1 to 5). With no loss of interpretability, the tic marks and tic labels have been purposely omitted for conciseness. For a given material, the limits of the vertical axis are the same for all 20 plots so as to assist comparability. For example, the vertical axis limits for all plots for material 1 (Figure 19) are 0.040 W/m·K to 0.045 W/m·K. The horizontal limits change depending on what factor is plotted. For example, in the first plot of Figure 19, the horizontal axis varies from 1 to 5; whereas, in the fifth plot (λ versus plate size), there are three horizontal axis values (300 mm, 610 mm, and 1016 mm as taken from Table 4).

From an interpretation point of view, participating laboratories are encouraged to peruse each plot and relate their own laboratory values of λ to specific factors. By way of example (using Figure 19 for material 1 as a starting point for illustrative purposes), laboratory 1 appears to be high relative to the other four laboratories. It is of use to consider what laboratory factors may contribute to this high set of observations. In this regard, it is desirable to enumerate those factors in which laboratory 1 is unique, i.e., in which no other laboratories share that factor. Hence, for example, laboratory 1 has the following unique settings:

1) Plot 3: surface measurement technique is 1 (affixing thermocouples to specimen);
2) Plot 4: lowest ρ;
3) Plot 5: smallest plate size;
4) Plot 6: smallest meter plate size;
5) Plot 13: lowest cold plate temperature (T_c);
6) Plot 17: smallest Q; and,
7) Plot 18: smallest meter area (A).

Note that the above items 3), 4), and 7) all share the same common dimensionality. The net effect is that the above factors serve to verify previous conclusions.

On the other hand, perusal of certain plots would suggest that other factors would not serve as an explanation for the high observations reported by laboratory 1. Thus, any future settings shared with laboratory 1 would suggest that the factor might be eliminated as a prime candidate for causing high values of λ. For example, laboratories 1 and 4 both having the same plate emittance (plot 7) suggests exclusion, as would laboratories 1, 2, 3, and 5 sharing the same (double-sided) mode of operation (plot 11).

Figures 19 through 22 are replete with information dealing with both consistency and anomaly. Participant laboratories are encouraged to use Figures 19 to 22 (and the upcoming Tables 18 and 19) for self-assessment.

6.3.1 Analysis of Variance: Question 1 from this section can be re-phrased: Do different levels of a factor yield significantly different λ's from one another. The analysis of variance (ANOVA) procedure examines the statistical significance of a factor on λ. The term *FCDF* (F-cumulative distribution function) given in Figures 19 through 22 is the percent point of the F-distribution

[16, p. 117]; only *FCDF* values above 95 % are considered significant (at the 5 % level). It is important to note that values of *FCDF* are based on the assumption that the variances of the treatments[3] are constant across treatments. This is decidedly not the case for many plots and so the numeric values of *FCDF* should be used for indicative purposes only. An advantage of the ANOVA analysis is that it is applicable to both types of data: quantitative (numeric) or qualitative (categorical). Table 18 summarizes the results of the ANOVA procedure.

TABLE 18 – Is a Factor Statistically Significant? (*FCDF* > 95 %? Yes/No)

	Laboratory Factors	Material 1	Material 2	Material 3	Material 4
0	Laboratory (primary)	Y	Y	Y	N
1	Steady-state conditions	Y	Y	Y	N
2	Conditioning of specimen	Incomplete	Incomplete	Incomplete	Incomplete
3	Measurement technique for surface temperatures	Y	Y	N	Y
4	Bulk density (ρ)	Y	Y	Y	N
5	Plate size	Y	Y	N	N
6	Meter plate size	Y	Y	Y	N
7	Plate emittance	Y	Y	Y	N
8	Type of heater	N	Y	N	N
9	Edge guarding	Y	N	Y	N
10	Temperature sensor	N	Y	Y	Y
11	Operation mode	N	N	Y	N
12	T_h	Y	N	Y	Y
13	T_c	Y	N	Y	Y
14	T_m	Y	N	Y	Y
15	ΔT	Y	Y	Y	Y
16	L	N	Y	Y	N
17	Q	Y	Y	Y	N
18	A	Y	Y	Y	N
19	Q	Y	Y	Y	N

From Table 18, the single most important conclusion is that, for material 4, the primary factor laboratory is *not* statistically significant. This is not the case for materials 1, 2, and 3 – there is statistically significant difference across the 5 laboratories.

Further examination of Table 18 above indicates that many of the 19 (secondary) laboratory factors are significant. Significance, however, does not necessarily imply causation – especially given the fact that many correlations exist among the factors themselves. (For example, if T_h is significant and/or T_c is significant, then it is not surprising that T_m and/or ΔT would also be significant.) Finding the root significant factor(s) is done by using results from Table 18 in conjunction with engineering judgement (and possibly additional tests) by the participating laboratories.

6.3.2 Correlation: Question 2 from this section deals with relationships between λ and a factor. The simplest case, asks whether a linear relationship exists between λ and a factor. The correlation coefficient (r) is a measure of linear relatedness between two variables. A perfect

[3] A treatment is a particular combination of levels of the factors involved in an experiment [17].

linear relationship yields a correlation coefficient of ± 100 %. A lack of relationship yields a correlation coefficient of zero (0 %). Because of the large quantity of replicate data for the five laboratories (n = 100 per plot), values of $|r|$ > 20 % are statistically significant at the 5 % level [16, p. 185 and p. 557].

In the context of linear regression between λ and a factor, the correlation coefficient is related to the slope of the fitted line. If the slope is positive (or negative), the correlation coefficient will be positive (or negative). If the slope is zero, the correlation coefficient will be zero.

Table 19 summarizes the results of the correlation analysis. In cases where the data for the factor on the x-axis is qualitative (as identified in Table 13), the correlation coefficient is meaningless and was not included. Values of r with a "high" level of correlation (arbitrarily selected for $|r|$ > 90 %) are shown in ***boldface italics***.

TABLE 19 – Is there a Significant Correlation between λ and a Laboratory Factor? ($|r|$ >20 %? Yes/No)

	Laboratory Factors	Material 1	Material 2	Material 3	Material 4
0	Laboratory (primary)	---	---	---	---
1	Steady-state conditions	---	---	---	---
2	Conditioning of specimen	Incomplete	Incomplete	Incomplete	Incomplete
3	Measurement technique for surface temperatures	---	---	---	---
4	Bulk density (ρ)	***Y (−98 %)***	Y (+63 %)	N	Y (+30 %)
5	Plate size	Y (−76 %)	Y (−89 %)	N	Y (−29 %)
6	Meter plate size	Y (−77 %)	***Y (−94 %)***	Y (−31 %)	Y (−41 %)
7	Plate emittance	Y (+36 %)	Y (+44 %)	Y (−47 %)	Y (+25 %)
8	Type of heater	---	---	---	---
9	Edge guarding	---	---	---	---
10	Temperature sensor	---	---	---	---
11	Operation mode	---	---	---	---
12	T_h	Y (−51 %)	N	Y (+82 %)	Y (+27 %)
13	T_c	Y (−73 %)	Y (−70 %)	Y (−43 %)	Y (−26 %)
14	T_m	Y (−62 %)	Y (−53 %)	Y (+36 %)	N
15	ΔT	Y (−33 %)	Y (+46 %)	Y (+73 %)	Y (+34 %)
16	L	N	Y (−49 %)	N	Y (+39 %)
17	Q	Y (−47 %)	Y (−75 %)	N	Y (−42 %)
18	A	Y (−76 %)	***Y (−94 %)***	Y (−35 %)	Y (−44 %)
19	Q	Y (+69 %)	Y (+43 %)	Y (+53 %)	N

The values given in Table 19 indicate that for all materials there are several factors correlated with λ. Fortunately, many of the numeric values of $|r|$ are small. For materials 1 and 2, however, there are a few factors highly correlated with λ (> 90 %) that call for further investigation. Note that high correlation does *not* imply causality, only a linear association. In general, these results are somewhat unexpected and indicate that future interlaboratory comparisons should probably require additional protocols to minimize the effects of undesired laboratory factors.

6.4 Comparison with Certified Values

The comparison of measured values of λ with certified values, $\lambda(T_m, \rho)$, provides another useful method for evaluating the differences among the laboratories from an engineering perspective. Values of $\lambda(T_m, \rho)$ for materials 1 and 4 (SRMs 1451 and 1453, respectively) were determined from Eq 1 using the measured values of T_m and ρ provided by each participant (Table 8). The corresponding uncertainties of ± 3 % and ± 1.3 % for materials 1 and 4, respectively, were used to establish cut-off bounds for the evaluation of anomalous values of λ reported earlier. The certification equation for material 2 [10] became available after this analysis was completed and is not included. Material 3 is currently undergoing certification process and, therefore, the certification equation is unavailable.

Table 20 summarizes the measurements for materials 1 and 4 given previously in Table 8 for ρ, T_m, ΔT, λ, as well as computed values of $\lambda(T_m, \rho)$ from Eq 1, and the corresponding differences (absolute and relative) between λ and $\lambda(T_m, \rho)$. The data are partitioned by material, laboratory, and replicate number (i.e., laboratory run sequence). For materials 1 and 4, the relative differences range from approximately −0.2 % to +8.5 % and −0.2 % to +2.4 %, respectively. Figure 23 plots the relative differences of λ and $\lambda(T_m, \rho)$ versus ρ for materials 1 and 4, respectively. Here the plot character represents the laboratory and horizontal solid lines indicate the cut-off bounds for the (expanded) uncertainty levels for each CRM. For materials 1 and 4, the (expanded) uncertainties are ± 3 % (coverage factor unavailable) and ± 1.3 % (coverage factor of $k = 2$), respectively.

Figure 23a reveals that the relative differences for the replicate data from laboratories 2, 3, 4, and 5 are entirely within the uncertainty levels (± 3 %) for material 1. With the exception of one observation from laboratory 4, the differences from the certified values for these laboratories are less than 1.3 %. Although the agreement among these laboratories is quite encouraging, the differences for laboratory 1 are considerably outside the uncertainty levels for SRM 1451. For material 1, the data from laboratory 1 are considered sufficiently different (+7.2 % to +8.5 %) from the certified values to warrant rejection as an outlying observation.

Figure 23b reveals that the relative differences for the replicate data from laboratories 2, 3, 4, and 5 are entirely within the uncertainty levels (± 1.3 %) for material 4. For these laboratories, the differences from the certified values are less than 0.8 %. Although the agreement among these laboratories is quite encouraging, the differences for laboratory 1 require further analysis. For laboratory 1, 3 of the 5 replicate observations, including the mean value, from laboratory 1 are within the uncertainty levels. Therefore, the differences for laboratory 1 are considered acceptable and within the CRM uncertainty levels for material 4. In general, the agreement among the laboratories for material 4 (SRM 1453) is strongly encouraging.

TABLE 20 – Comparison of Fixed Temperature (297.15 K) Replicate Data with Certified Values

Material	Lab	Replicate	ρ (kg/m^3)	T_m (K)	ΔT (K)	λ (W/m·K)	$\lambda(T_m,\rho)$ (W/m·K)	Difference (W/m·K)	Difference (%)
1	1	1	13.35	296.97	20.47	0.04473	0.04138	0.00335	8.09
1	1	2	13.35	296.88	20.27	0.04489	0.04136	0.00353	8.54
1	1	3	13.35	294.88	16.57	0.04425	0.04088	0.00337	8.24
1	1	4	13.35	294.89	16.69	0.04417	0.04088	0.00329	8.04
1	1	5	13.35	296.87	20.66	0.04434	0.04136	0.00299	7.22
1	2	1	14.10	297.16	19.96	0.04101	0.04064	0.00037	0.90
1	2	2	14.10	297.17	20.01	0.04104	0.04064	0.00040	0.97
1	2	3	14.10	297.19	20.01	0.04105	0.04065	0.00040	0.99
1	2	4	14.10	297.20	20.00	0.04103	0.04065	0.00038	0.93
1	2	5	14.10	297.20	19.98	0.04109	0.04065	0.00044	1.08
1	3	1	14.15	297.15	20.00	0.04052	0.04059	-0.00007	-0.18
1	3	2	14.15	297.15	20.00	0.04055	0.04059	-0.00004	-0.10
1	3	3	14.15	297.15	20.00	0.04056	0.04059	-0.00003	-0.08
1	3	4	14.15	297.15	20.00	0.04056	0.04059	-0.00003	-0.08
1	3	5	14.15	297.15	20.00	0.04056	0.04059	-0.00003	-0.08
1	4	1	14.13	297.01	18.93	0.0415	0.04058	0.00092	2.27
1	4	2	14.13	297.10	19.06	0.0411	0.04060	0.00050	1.23
1	4	3	14.13	297.12	19.07	0.0411	0.04061	0.00049	1.22
1	4	4	14.13	297.11	19.07	0.0411	0.04060	0.00050	1.22
1	4	5	14.13	297.09	19.03	0.0411	0.04060	0.00050	1.24
1	5	1	14.37	297.16	19.98	0.04035	0.04039	-0.00004	-0.10
1	5	2	14.37	297.15	20.02	0.04030	0.04039	-0.00009	-0.21
1	5	3	14.37	297.14	20.01	0.04029	0.04038	-0.00009	-0.23
1	5	4	14.37	297.14	19.96	0.04031	0.04038	-0.00007	-0.18
1	5	5	14.37	297.14	19.97	0.04034	0.04038	-0.00004	-0.11
4	1	1	39.8	296.15	20.70	0.03401	0.03346	0.00055	1.66
4	1	2	39.8	296.67	20.51	0.03433	0.03352	0.00081	2.41
4	1	3	39.8	295.70	21.02	0.03350	0.03341	0.00009	0.26
4	1	4	39.8	295.97	20.82	0.03382	0.03344	0.00038	1.13
4	1	5	39.8	296.19	20.77	0.03390	0.03346	0.00044	1.30
4	2	1	38.6	297.15	20.00	0.03369	0.03363	0.00006	0.19
4	2	2	38.6	297.14	20.05	0.03369	0.03363	0.00006	0.19
4	2	3	38.6	297.16	19.96	0.03372	0.03363	0.00009	0.27
4	2	4	38.6	297.14	20.01	0.03368	0.03363	0.00005	0.16
4	2	5	38.6	297.18	20.04	0.03368	0.03363	0.00005	0.15
4	3	1	38.2	297.15	20.00	0.03367	0.03364	0.00003	0.08
4	3	2	38.2	297.15	20.00	0.03378	0.03364	0.00014	0.40
4	3	3	38.2	297.15	20.00	0.03377	0.03364	0.00013	0.37
4	3	4	38.2	297.15	20.00	0.03376	0.03364	0.00012	0.34
4	3	5	38.2	297.15	20.00	0.03377	0.03364	0.00013	0.37
4	4	1	38.8	296.78	17.76	0.0335	0.03357	-0.00007	-0.22
4	4	2	38.8	296.78	17.82	0.0338	0.03357	0.00023	0.67
4	4	3	38.8	296.84	17.87	0.0338	0.03358	0.00022	0.65
4	4	4	38.8	296.79	17.80	0.0337	0.03358	0.00012	0.37
4	4	5	38.8	296.78	17.79	0.0336	0.03357	0.00003	0.08
4	5	1	38.5	297.13	19.99	0.03387	0.03363	0.00024	0.72
4	5	2	38.5	297.15	19.97	0.03386	0.03363	0.00023	0.68
4	5	3	38.5	297.13	19.98	0.03386	0.03363	0.00023	0.68
4	5	4	38.5	297.14	19.99	0.03389	0.03363	0.00026	0.77
4	5	5	38.5	297.13	20.00	0.03388	0.03363	0.00025	0.74

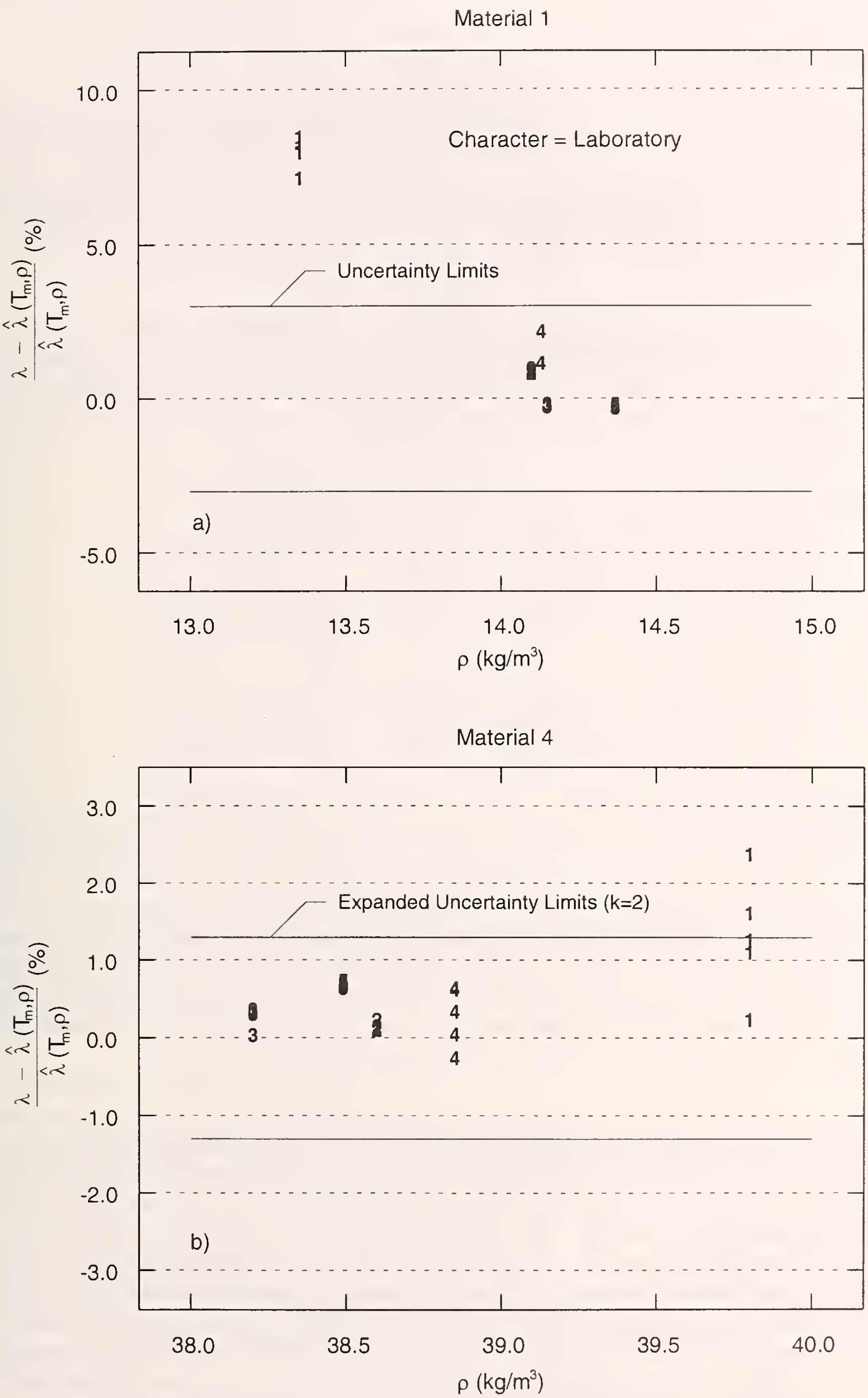


Figure 23. Relative deviations from certified values versus ρ (Materials 1, 4)

6.5 Comparison with Standard Test Method Precision Indices

Both ISO 8302 [2] and ASTM C 177 [3] provide statements of uncertainty (precision) of ± 2 % for measurements near room temperature. In addition, ISO 8302 [2] specifies a reproducibility[4] of better than ± 1 % for independent replicate measurements near room temperature. This section evaluates the question: Are the differences among laboratories at 297.15 K significant in comparison with accepted uncertainty and precision statements of the standard test methods for guarded-hot-plate apparatus [2,3]?

From previous analyses, two sets of laboratory data are sufficiently different to warrant rejection as outlying observations – data sets for material 1, laboratory 1 (5 observations); and, material 3, laboratory 4 (5 observations). Table 21 excludes these observations and re-computes the laboratory relative means and the grand relative standard deviations previously given in Table 11 for the affected materials.

TABLE 21 – Relative Means and Standard Deviations for Replicates (297.15 K) Excluding Outlying Data (Material 1-Lab1 and Material 3-Lab 4)

Lab	Material 1		Material 2		Material 3		Material 4	
	Mean (%)	SD (%)	Mean (%)	SD (%)	Mean (%)	SD (%)	Mean (%)	SD (%)
1	---	---	1.4	0.16	−0.1	1.19[1]	0.39	0.89[1]
2	0.7	0.07	−0.6	0.06	0.5	0.04	−0.26	0.05
3	−0.5	0.04	−1.3	0.11	−1.1	0.47[2]	−0.09	0.13
4	1.0	0.43[2]	0.0	0.17	---	---	−0.30	0.39[2]
5	−1.1	0.06	0.4	0.56[1]	0.8	0.11	0.27	0.04
Grand	---	0.91	---	0.95	---	0.95	---	0.49
Range	1.8	---	2.7	---	1.9	---	0.69	---

[1]High; [2]Marginally high

From Table 21, note that the ranges of laboratory means for materials 1, 2, 3, and 4 are 1.8 %, 2.7 %, 1.9 %, and 0.69 %, respectively. The corresponding half-ranges for materials 1, 2, 3, and 4 are ± 0.9 %, ± 1.4 %, ± 1.0 %, and ± 0.35 %, respectively, which are all less than the ISO/ASTM uncertainty (precision) statements of ± 2 %. With the exception of one set of data (material 3, laboratory 1), the laboratory standard deviations are all less than 1 % (Table 21), which are less than the reproducibility limit of ± 1 % given in ISO 8302 [2].

At this point, it is constructive to re-compute the summary statistics for the replicate data, excluding the above outlying observations (material 1, laboratory 1 and material 3, laboratory 4). Tables 22a and 22b exclude these observations and re-present the laboratory means and grand standard deviations previously given in Tables 10a and 10b, respectively. The last row in each table provides the recomputed values for the respective grand or "pooled" statistic for each material (across all laboratories). The last column in each table provides the recomputed values for the respective grand or "pooled" statistic for each laboratory (across all materials). As noted in the last row of Table 22a, the grand mean for material 1 has decreased and increased for material 3 when compared to previous values (Table 10a). Values for the grand standard deviations (last row, Table 22b) have decreased for materials 1 and 3. The 'pooled" values for the lab standard deviations have decreased for materials 1 and increased slightly for material 3.

[4] ASTM defines this quantity as repeatability.

TABLE 22a – Means for Replicate Data (297.15 K)
Excluding Outlying Data (Material 1-Lab1 and Material 3-Lab 4)

Lab	Material 1	Material 2	Material 3	Material 4	Lab
	$\bar{\lambda}$ (W/m K)	$\bar{\lambda}$ (W/m K)	$\bar{\lambda}$ (W/m K)	$\bar{\lambda}$ (W/m K)	Average (W/m K)
1	---	0.03251	0.03655	0.03391	---
2	0.04104	0.03189	0.03675	0.03369	0.03584
3	0.04055	0.03166	0.03616	0.03375	0.03553
4	0.04118	0.03206	---	0.03368	---
5	0.04032	0.03220	0.03686	0.03387	0.03581
Grand	0.04077	0.03206	0.03658	0.03378	0.03591

TABLE 22b – Standard Deviations for Replicate Data (297.15 K)
Excluding Outlying Data (Material 1-Lab1 and Material 3-Lab 4)

Lab	Material 1	Material 2	Material 3	Material 4	Pooled
	SD (λ) (W/m K)	SD (λ) (W/m K)	SD (λ) (W/m K)	SD (λ) (W/m K)	SD (W/m K)
1	---	0.00005	0.00043	0.00030	0.00031
2	0.00003	0.00002	0.00002	0.00002	0.00002
3	0.00002	0.00004	0.00017	0.00005	0.00009
4	0.00018	0.00005	---	0.00013	0.00013
5	0.00003	0.00018	0.00004	0.00001	0.00009
Pooled	0.00009	0.00009	0.00023	0.00015	0.00016
Grand	0.00037	0.00030	0.00035	0.00017	---

Figure 24 re-plots both the "pooled" (within-laboratory) and grand standard deviations (within- and between-laboratory) (last two rows from Table 22b, respectively) versus the grand means (Table 22a) of the thermal conductivities for materials 1 through 4. Each data point is depicted by a plot character equal to material. The results of Figure 22 indicate that the between-laboratory variability is approximately the same across all levels of thermal conductivity. This result is reassuring for the four materials studied in this comparison. The within-laboratory variability, however, is approximately the same across all levels of thermal conductivity and is, as expected, less than the between-laboratory variability for each material.

7 Summary Findings for Replicate Data (297.15 K)

This section summarizes the important findings of the replicate data analysis. The principal conclusion from the comparison of the replicate data is that the behavior of the laboratories does, in fact, change from material to material. As observed initially in Figure 4, the location and, to a lesser extent, variation of each set of laboratory data does change from material to material. In short, there is a laboratory-material interaction. This conclusion was verified by a re-examination of the data that included the engineering levels of expanded uncertainty for each laboratory (Figure 5). Subsequent analyses were guided by a set of primary questions (formulated from the results of Figure 4) that represent the core results of this interlaboratory comparison.

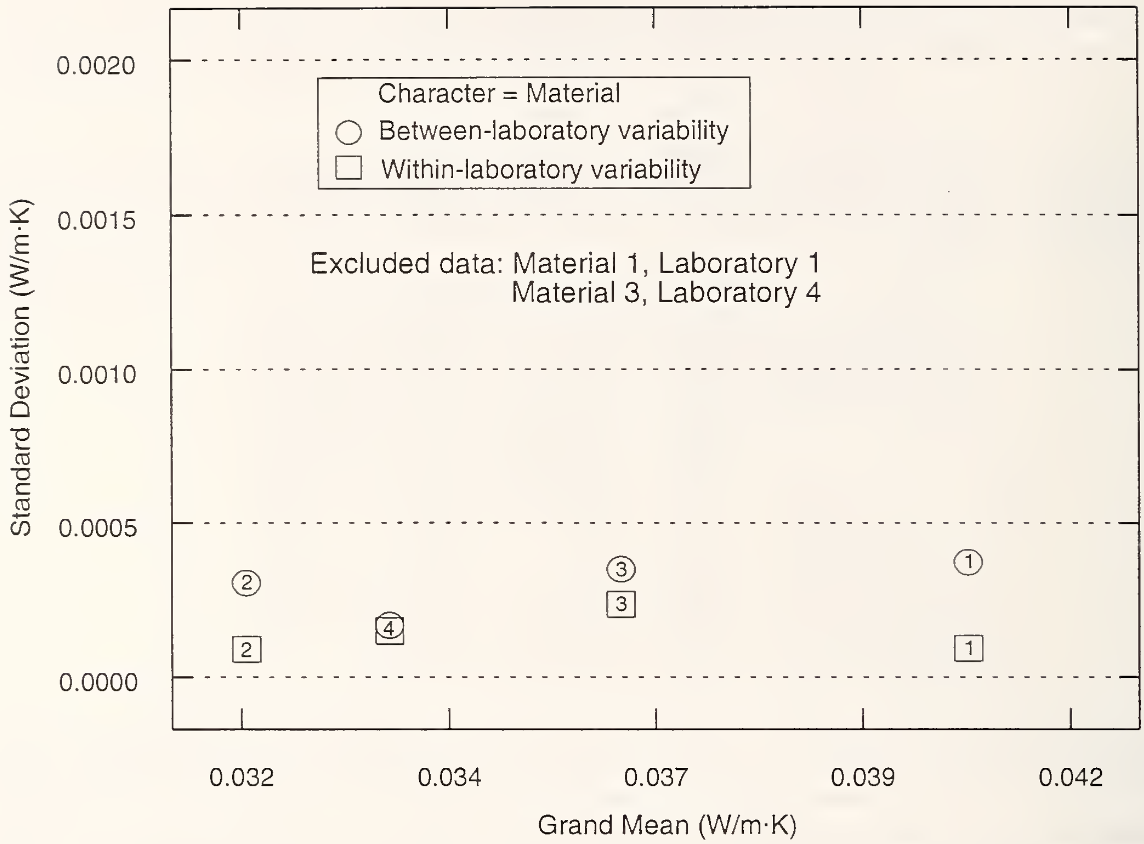


Figure 24. Within- and between-laboratory variability versus grand means (Materials 1, 2, 3, 4) Excluding data from Material 1, Laboratory 1 and Material 3, Laboratory 4

7.1 Descriptive Statistics

Descriptive statistics are used to summarize the fixed-temperature (297.15 K) replicate data and to subsequently rank order the laboratories and materials. The results of this detailed statistical analysis quantify the changes in location and variation (observed in Figures 4 and 5). These results, which are drawn from Tables 10 and 11, are summarized below using a shorthand notation (M = material, L = laboratory). The changes in location were greatest for material 1, a fibrous-glass blanket and smallest for material 4, a semi-rigid expanded polystyrene board.

- Laboratory location rank by material (relative mean in parentheses) from Table 11:
  - M1: L1 (7.1 %), L4 (−0.8 %), L2 (−1.1 %), L3 (−2.3 %), L5 (−2.9 %);
  - M2: L1 (1.4 %), L5 (0.4 %), L4 (0.0 %), L2 (−0.6 %), L3 (−1.3 %);
  - M3: L5 (1.6 %), L2 (1.3 %), L1 (0.8 %), L3 (−0.3 %), L4 (−3.5 %); and,
  - M4: L1 (0.39 %), L5 (0.27 %), L3 (−0.09 %), L2 (−0.26 %), L4 (−0.30 %).

- Laboratory variation rank by material (relative standard deviation in parentheses) from Table 11:
  - M1: L1 (0.71 %), L4 (0.43 %), L2 (0.07 %), L5 (0.06 %), L3 (0.04 %);
  - M2: L5 (0.56 %), L4 (0.17 %), L1 (0.16 %), L3 (0.11 %), L2 (0.06 %);
  - M3: L1 (1.19 %), L3 (0.47 %), L5 (0.11 %), L2 (0.04 %), L4 (0.0 %); and,
  - M4: L1 (0.89 %), L4 (0.39 %), L3 (0.13 %), L2 (0.05 %), L5 (0.04 %)

- Material variation rank (grand relative standard deviation in parentheses) from Table 11:
  - M1 (3.74 %), M3 (1.97 %), M2 (0.95 %), and M4 (0.49 %)

- Anomalous data points:
  - High location and variation: (M1, L1)
  - High variation: (M2, L5); (M3, L1); and, (M4, L1)
  - Low location and artificially low variation: (M3, L4)

- Marginal data points:
  - Marginally high variation: (M1, L4); (M3, L3); and (M4, L4)

7.2 Assessment

The assessment of the fixed-temperature (297.15 K) replicate data was guided by the six core questions presented above. The systematic effects noted in the data (Figures 4 and 5) indicate the presence of one or more secondary factors. Applying a cause and effect diagram (Figure 12), (secondary) laboratory factors were identified and systematically investigated using statistical and engineering techniques. The results for questions 1 through 6 are presented below.

Q1: For 2 of the 4 materials, laboratory 1 is high, why? It is surmised that, for laboratory 1, much of the within-laboratory variability observed in the test data (Figures 4 and 5) could be attributed to the measurement technique of affixing thermocouples directly to the specimen for surface temperatures. In an early comparison of guarded hot plates, Robinson and Watson [15] noted that discrepancies could result between conductivity values obtained using temperatures from plate surfaces and those measured using surface thermocouples. This technique introduces several potential sources of variation to the temperature difference across the specimen:

1) operator/procedural judgement in precisely locating the sensor for each replicate;
2) relocation of sensor could place the sensor in different proximity to local material inhomgenieities;
3) different levels of clamping pressure could result in temperature variations if the thermocouples were not in intimate contact with the plates; and,
4) (potential) error due to incorrect determination of the separation distance corresponding to the temperature difference across the specimen.

With the above concerns, one could make an effective argument that this technique would seem to have inherently more variation than sensors permanently affixed to the apparatus (as utilized by the other laboratories) and, thus, contribute to less precise levels of replication. This premise was investigated by plotting the laboratory test results for λ on a relative basis (%) versus material (Figure 13). For laboratory 1, the relative differences from the grand mean are substantially different across the four materials (Figure 13b) and the relative standard deviations are high for materials 1, 3, and 4 (Figure 13c). Materials 1, 2, and 3 are fibrous materials having average bulk densities of 14 kg/m^3, 72 kg/m^3, and 227 kg/m^3, respectively. Material 1 is a flexible blanket and materials 2 and 3 are semi-rigid boards of increasing rigidity. Material 4 is a semi-rigid expanded polystyrene board having relatively smooth surfaces in comparison to the fibrous materials. The results in Figure 13 would tentatively suggest an interaction between the surface measurement technique of affixing the sensors to the materials and the type of material.

Q2: For material 1, laboratory 5 is low, why? Referring to Figure 14, there is, unfortunately, a small density effect for specimen sample that was distributed. The value for λ is low because the bulk density of the specimen was high.

Q3: For material 2, laboratory 3 is low, why? The only insight to this finding is given in Figure 20, which indicates an extremely high negative correlation between λ and meter plate size (as well as the two other related factors). The reader is strongly cautioned, however, that correlation does not imply causality.

Q4: For material 3, laboratory 4 is low, why? The answer was provided by laboratory 4 in their official report form. In the comments section of their official test report form, laboratory 4 reported that, "this material had completely delaminated on arrival so that the test specimen consisted of two pieces which were always aligned in the same orientation with respect to each other whilst testing."

Q5: For 3 of the 4 materials, laboratory 1 is noisy, why? The answer is given above (see question 1).

Q6: For material 2, laboratory 5 appears to have one outlying observation, why? The answer to this question is currently unknown.

7.2.1 Anomalous Data Points: Analyses of the anomalous data points above revealed that two sets of data points (10 % of the replicate data) were outlying data sets (and eventually omitted). For interlaboratory studies, it is extremely helpful (and inevitably necessary) for the laboratories in question to present their own explanations for the behavior of the test results. To their credit, laboratories 1 and 4 did provide explanations (see below) for their anomalous data.

M1, L1: After submission of their test data, laboratory 1 reported that the surface temperatures for determinations of specimen ΔT were measured using 0.2 mm diameter thermocouples placed directly on the surface of the specimen with adhesive tape. In contrast, the other laboratories utilized temperature sensors permanently mounted in the heating and cooling surfaces. Further, this set of data was significantly different (by 8 %) from certified values for material 1 and therefore classified as an outlying data set. For material 1, all of other laboratory data was entirely within the bounds of the CRM uncertainty limits of ± 3 %.

M3, L4: As reported by laboratory 3, the reason this data set was identified as an outlying set data was attributed to specimen failure. The specimen was found delaminated upon arrival. No other laboratory reported this condition for the same material. As a side comment, this one incident of delamination could present a dilemma for future "certification" of material 3. One occurrence can be attributed as an accident (i.e., handled badly during shipment, etc). If the occurrences are more regular, the material may be unsuitable as a reference material.

Two of the other sets of anomalous data points (M3, L1 and M4, L1) were attributed to the technique used to measure the temperature difference across the specimens (see Question 1, above). The underlying cause(s) for the last set of anomalous data points (M2, L5) are undetermined.

7.2.2 Marginal Data Points: The underlying cause(s) for the marginal data points identified in the statistical analysis are unknown.

7.2.3 Laboratory Factors: The contribution of any (secondary) laboratory factors was systematically investigated using a cause-and-effect diagram. The analysis was covered in two parts: an engineering assessment of the major sources of variation that included a check of the protocol execution; and, a comprehensive statistical analysis of 19 laboratory factors using correlation analysis and analysis of variance (ANOVA).

Check of Protocol Execution:

- Checking the execution of the protocol revealed that, for some laboratories, there were deviations in obtaining the target temperatures for the hot and cold surfaces. As a result the mean temperature (T_m) and temperature differences (ΔT) were slightly different than the target temperatures for some laboratories.

- For the report values of q (heat flux), two laboratories reported values that were essentially twice the values of the other laboratories for a given material. This was merely a bookkeeping discrepancy and did not affect the final determination of thermal conductivity. Still, this discrepancy indicates that additional clarification is needed in the standard test methods [2,3] concerning the definition of heat flux and what quantity is to be reported.

7.2.4 Comparison of λ with Certified Values: Certified values were available for materials 1 and 4, fibrous-glass blanket (SRM 1451) and expanded polystyrene board (SRM 1453), respectively. For material 1, the mean values of λ for 4 of the 5 laboratories deviated from certified values by less than the ± 3 % uncertainty limits. For material 4, the mean values of λ for 5 of the 5 laboratories were less than the ± 1.3 % relative expanded uncertainties ($k = 2$).

7.2.5 Comparison with Standard Test Methods Precision Indices: When the outlying data sets are excluded (for the reasons noted above), the ranges of laboratory means for materials 1, 2, 3, and 4 are 1.8 %, 2.7 %, 1.9 %, and 0.69%, respectively (Table 21). The corresponding half-ranges for materials 1, 2, 3, and 4 are ± 0.9 %, ± 1.4 %, ± 1.0 %, and ± 0.35 %, respectively, which are all less than the ISO/ASTM uncertainty (precision) statements of ± 2 %. With the exception of one set of data (material 3, laboratory 1), the laboratory standard deviations are all less than 1 % (Table 21), which are all less than the reproducibility limit of ± 1 % given in ISO 8302 [2].

7.2.6 Procedural Recommendations for Future Comparisons:

- It would be useful to have additional information on the measurement technique(s) used by individual laboratories participating in the comparison (for example, in-situ techniques for thickness measurements, etc). In a related issue, the list of factors investigated here was not exhaustive. For example, other factors that could be important are: 1) major differences in gap size between metering and guard and how the gap was filled; 2) what areas were used to calculate λ if, in fact, there were gap width differences; and, 3) how well was edge guarding undertaken – could moisture condensation be a factor? A standardized list of requested factors would be extremely useful.

- One laboratory utilized an alternative technique for the measurement of the temperature difference across the specimen. The results suggest that this technique of placing temperature sensors directly on the specimens is inherently less precise than using sensors permanently mounted in the heating and cooling surfaces. Both ISO and ASTM may wish to address this issue with greater clarity in their respective standards [2,3].

- The five laboratories used slightly different criteria for establishing settling and equilibrium times for taking test data. Both ISO and ASTM may wish to address this issue in their respective standards [2,3].

- Two of the laboratories computed values of q approximately one-half the values determined by the other three laboratories. Both ISO and ASTM may wish to address this issue in their respective standards [2,3].

- In future comparisons, laboratories should follow an agreed upon (or standardized) method for computing the expanded uncertainties (U) of their measured values of thermal conductivity (λ).

- In order to minimize some of these procedural differences in the future, the respective ISO and ASTM technical committees may wish to develop standard practices for conducting an interlaboratory comparison specifically for guarded hot plate apparatus. These standard practices should include standard test protocols, forms for reporting data (including expanded uncertainties (U)), and ancillary information (such as test equipment, etc.). Such activities will need to consider that ISO and ASTM have different definitions for some statistical terms, such as reproducibility and repeatability.

8. Analysis of Multi-Temperature (280 K to 320 K) Data

The third primary factor investigated in this interlaboratory comparison is temperature, specifically, single-point measurements for each material from 280 K to 320 K. The single-point data precludes the rigorous descriptive statistical analysis performed for the replicate data at 297.15 K. Nonetheless, the analyses are driven by the same central theme for the fixed-temperature replicate data: How do the laboratories behave across the four materials? With respect to this question, the following analyses are provided:

1) Presentation of laboratory data;
2) Graphical exploration of laboratory test data versus temperature by material;
3) An ensuing ranking of the laboratories by location and variation;
4) Comparison of the thermal conductivity measurements with certified values; and,
5) Comparison of the thermal conductivity measurements with the precision indices from standard test methods [2,3]

8.1 Presentation of Laboratory Data

Table 23 summarizes the measurements for the specimen bulk density (ρ), hot (T_h) and cold (T_c) plate temperatures, specimen heat flow (Q), thickness (L), meter area (A), thermal conductivity (λ), mean temperature (T_m), and temperature difference (of 20 K). The tabulated data are partitioned by material, laboratory, and run sequence. All laboratories, except 4, reported a run sequence from low to high T_m. Note that for all materials, laboratory 4 did not report data at 320 K. The value for ρ has been rounded to 3 significant digits and represents the average of the pair of specimens (for a double-sided test). The number of significant digits for the other parameters is tabulated as received from the laboratory. Any certainty for the 5th significant digit for values of λ cannot be assigned, but the digit is included for the subsequent analyses of the results. The data in Table 23 were entered in an electronic spreadsheet and reported values for λ were checked by recalculation.

8.2 Graphical Exploration of Laboratory Data

Figure 25 plots the measurements of λ versus T_m for the 4 materials. The observations for each laboratory are shown as characters 1, 2, 3, 4, and, 5, respectively. In examining the laboratory data for each material, the central question under investigation is again, do the five laboratories behave similarly across the four materials? Or, (if the laboratories behave differently across the four materials), is there a laboratory-material interaction? There are two independent but related questions in determining the behavior of laboratories from material to material: 1) Is there a change in location of the laboratory data? and, 2) Is there a change in variation of the laboratory data? Examination of these two questions for the data in Figure 25 is provided by a linear regression analysis of the data. For comparison purposes, the data in Figure 25 were fit with the following model:

$$\lambda = b_0 + b_1 T_m \qquad (3)$$

Figure 26 plots the relative deviations from the fitted curve for each data point.

TABLE 23 – Multi-Temperature (280 K to 320 K) Data

Material	Lab	Run Seq.	ρ (kg/m³)	T_h (K)	T_c (K)	Q (W)	L (W)	A (m²)	λ (W/m K)	T_m (K)	ΔT (K)
1	1	1	13.4	290.22	270.48	1.441	25.4	0.0225	0.04120	280.35	19.74
1	1	2	13.4	299.95	279.90	1.526	25.4	0.0225	0.04296	289.92	20.05
1	1	3	13.4	309.89	289.68	1.641	25.4	0.0225	0.04583	299.79	20.21
1	1	4	13.4	320.30	299.77	1.785	25.4	0.0225	0.04908	310.03	20.53
1	1	5	13.4	329.35	309.69	1.849	25.4	0.0225	0.05309	319.52	19.66
1	2	1	14.1	290.17	270.07	5.282	25.40	0.090016	0.03707	280.12	20.10
1	2	2	14.1	300.14	280.09	5.585	25.40	0.090056	0.03929	290.12	20.05
1	2	3	14.1	310.16	290.19	5.911	25.40	0.090097	0.04171	300.18	19.97
1	2	4	14.1	320.19	300.17	6.292	25.40	0.090138	0.04428	310.18	20.02
1	2	5	14.1	330.13	310.21	6.649	25.40	0.090178	0.04700	320.17	19.92
1	3	1	14.2	290.15	270.14	3.720	25.52	0.1297	0.03656	280.15	20.01
1	3	2	14.2	300.15	280.15	3.9541	25.49	0.1297	0.03883	290.15	20.00
1	3	3	14.2	310.15	290.15	4.196	25.53	0.1298	0.04125	300.15	20.00
1	3	4	14.2	320.15	300.15	4.449	25.57	0.1299	0.04381	310.15	20.00
1	3	5	14.2	330.15	310.15	4.715	25.62	0.1299	0.04654	320.15	20.00
1	4	3	14.1	290.43	270.34	2.7613	25.38	0.09315	0.0374	280.39	20.09
1	4	4	14.1	300.26	280.16	2.9285	25.39	0.09315	0.0397	290.21	20.10
1	4	1	14.1	309.07	290.48	2.8712	25.42	0.09315	0.0422	299.77	18.59
1	4	2	14.1	319.72	300.44	3.1669	25.44	0.09315	0.0448	310.08	19.28
1	5	1	14.4	290.06	270.05	1.820	25.39	0.0625	0.03695	280.06	20.01
1	5	2	14.4	299.96	279.98	1.922	25.39	0.0625	0.03908	289.97	19.98
1	5	3	14.4	310.00	290.01	2.022	25.39	0.0625	0.04111	300.01	19.99
1	5	4	14.4	320.01	300.01	2.149	25.39	0.0625	0.04364	310.01	20.00
1	5	5	14.4	330.01	310.03	2.281	25.39	0.0625	0.04637	320.02	19.98
2	1	1	79.2	290.54	269.91	0.835	34.5	0.0225	0.03103	280.23	20.63
2	1	2	79.2	300.39	279.27	0.871	34.5	0.0225	0.03162	289.83	21.12
2	1	3	79.2	310.17	289.09	0.904	34.5	0.0225	0.03288	299.63	21.08
2	1	4	79.2	320.10	299.06	0.931	34.5	0.0225	0.03392	309.58	21.04
2	1	5	79.2	330.00	309.05	0.973	34.5	0.0225	0.03561	319.52	20.95
2	2	1	69.9	290.13	270.11	3.135	34.42	0.090015	0.02993	280.12	20.02
2	2	2	69.9	300.13	280.18	3.243	34.42	0.090056	0.03107	290.16	19.95
2	2	3	69.9	310.12	290.11	3.372	34.42	0.090097	0.03219	300.16	20.01
2	2	4	69.9	320.13	300.23	3.485	34.42	0.090137	0.03343	310.18	19.90
2	2	5	69.9	330.16	310.17	3.636	34.42	0.090178	0.03470	320.17	19.99
2	3	1	73.8	290.15	270.15	2.215	34.77	0.1297	0.02966	280.15	20.00
2	3	2	73.8	300.15	280.15	2.296	34.79	0.1297	0.03077	290.15	20.00
2	3	3	73.8	310.15	290.15	2.384	34.83	0.1298	0.03197	300.15	20.00
2	3	4	73.8	320.15	300.15	2.469	34.88	0.1299	0.03318	310.15	20.00
2	3	5	73.8	330.15	310.15	2.563	34.93	0.1299	0.03449	320.15	20.00
2	4	3	70.0	289.76	270.58	1.5659	34.36	0.09315	0.0301	280.17	19.18
2	4	4	70.0	300.14	280.40	1.6740	34.36	0.09315	0.0313	290.27	19.74
2	4	1	70.0	309.71	290.28	1.7071	34.39	0.09315	0.0324	300.00	19.43
2	4	2	70.0	319.69	300.14	1.7803	34.41	0.09315	0.0336	309.92	19.55
2	5	1	74.6	290.00	269.98	1.119	34.14	0.0625	0.03054	279.99	20.02
2	5	2	74.6	299.98	280.00	1.156	34.14	0.0625	0.03160	289.99	19.98
2	5	3	74.6	309.98	290.00	1.198	34.14	0.0625	0.03277	299.99	19.98
2	5	4	74.6	319.98	300.00	1.245	34.14	0.0625	0.03401	310.00	19.98
2	5	5	74.6	330.01	310.01	1.288	34.14	0.0625	0.03517	320.01	20.00

Material	Lab	Run Seq.	ρ (kg/m^3)	T_h (K)	T_c (K)	Q (W)	L (W)	A (m^2)	λ (W/m K)	T_m (K)	ΔT (K)
3	1	1	213	290.35	270.03	1.295	25.3	0.0225	0.03583	280.19	20.32
3	1	2	213	300.10	279.36	1.332	25.3	0.0225	0.03611	289.73	20.74
3	1	3	213	310.15	289.24	1.390	25.3	0.0225	0.03737	299.70	20.91
3	1	4	213	320.01	299.17	1.432	25.3	0.0225	0.03863	309.59	20.84
3	1	5	213	329.88	308.95	1.477	25.3	0.0225	0.03968	319.42	20.93
3	2	1	228	290.15	270.06	5.386	23.41	0.090015	0.03486	280.11	20.09
3	2	2	228	300.14	280.15	5.541	23.41	0.090056	0.03602	290.15	19.99
3	2	3	228	310.13	290.20	5.683	23.41	0.090097	0.03706	300.17	19.93
3	2	4	228	320.12	300.20	5.844	23.41	0.090137	0.03810	310.16	19.92
3	2	5	228	330.15	310.16	6.035	23.41	0.090178	0.03918	320.16	19.99
3	3	1	225	290.15	270.15	3.736	23.76	0.1297	0.03420	280.15	20.00
3	3	2	225	300.15	280.15	3.866	23.74	0.1297	0.03534	290.15	20.00
3	3	3	225	310.15	290.15	3.996	23.69	0.1298	0.03647	300.15	20.00
3	3	4	225	320.15	300.15	4.124	23.71	0.1299	0.03766	310.15	20.00
3	3	5	225	330.15	310.15	4.233	23.68	0.1299	0.03861	320.15	20.00
3	4	1	223	289.70	270.73	2.4387	24.00	0.09315	0.0331	280.21	18.97
3	4	2	223	299.31	280.54	2.4896	24.03	0.09315	0.0342	289.92	18.77
3	4	3	223	309.13	290.39	2.5607	24.05	0.09315	0.0353	299.76	18.74
3	4	4	223	319.07	300.26	2.6511	24.08	0.09315	0.0364	309.66	18.81
3	5	1	230	289.84	270.03	1.849	23.48	0.0625	0.0351	279.93	19.81
3	5	2	230	299.99	279.98	1.926	23.48	0.0625	0.03616	289.99	20.01
3	5	3	230	309.97	289.97	1.975	23.48	0.0625	0.03714	299.97	20.00
3	5	4	230	320.02	300.00	2.032	23.48	0.0625	0.03814	310.01	20.02
3	5	5	230	329.96	309.98	2.086	23.48	0.0625	0.03922	319.97	19.98
4	1	1	39.8	290.03	270.04	2.233	13.5	0.0225	0.03310	280.04	19.99
4	1	2	39.8	298.98	278.87	2.230	13.5	0.0225	0.03327	289.92	20.11
4	1	3	39.8	309.73	288.75	2.394	13.5	0.0225	0.03424	299.24	20.98
4	1	4	39.8	320.90	298.55	2.644	13.5	0.0225	0.03549	309.72	22.35
4	1	5	39.8	330.58	308.64	2.686	13.5	0.0225	0.03673	319.61	21.94
4	2	1	38.6	290.10	270.12	8.540	13.41	0.090015	0.03184	280.11	19.98
4	2	2	38.6	300.10	280.17	8.808	13.41	0.090056	0.03292	290.14	19.93
4	2	3	38.6	310.16	290.18	9.130	13.41	0.090097	0.03403	300.17	19.98
4	2	4	38.6	320.15	300.21	9.424	13.41	0.090138	0.03517	310.18	19.94
4	2	5	38.6	330.14	310.19	9.764	13.41	0.090178	0.03639	320.17	19.95
4	3	1	38.2	290.15	270.15	6.167	13.32	0.1297	0.03164	280.15	20.00
4	3	2	38.2	300.15	280.15	6.420	13.28	0.1297	0.03282	290.15	20.00
4	3	3	38.2	310.15	290.15	6.642	13.31	0.1298	0.03405	300.15	20.00
4	3	4	38.2	320.15	300.15	6.851	13.38	0.1299	0.03532	310.15	20.00
4	3	5	38.2	330.15	310.15	7.070	13.43	0.1299	0.03655	320.15	20.00
4	4	3	38.8	289.10	271.16	4.0270	13.12	0.09315	0.0316	280.13	17.94
4	4	4	38.8	298.96	281.08	4.1588	13.15	0.09315	0.0328	290.02	17.88
4	4	2	38.8	308.63	290.86	4.2638	13.17	0.09315	0.0339	299.75	17.77
4	4	1	38.8	318.73	300.92	4.4184	13.22	0.09315	0.0352	309.82	17.81
4	5	1	38.5	290.00	270.03	2.997	13.36	0.0625	0.03208	280.01	19.97
4	5	2	38.5	299.99	280.00	3.097	13.36	0.0625	0.03312	290.00	19.99
4	5	3	38.5	309.99	290.03	3.192	13.36	0.0625	0.03419	300.01	19.96
4	5	4	38.5	320.00	300.02	3.297	13.36	0.0625	0.03528	310.01	19.98
4	5	5	38.5	329.99	310.00	3.410	13.36	0.0625	0.03647	320.00	19.99

Note: Laboratory 4 did not report data at 320 K.

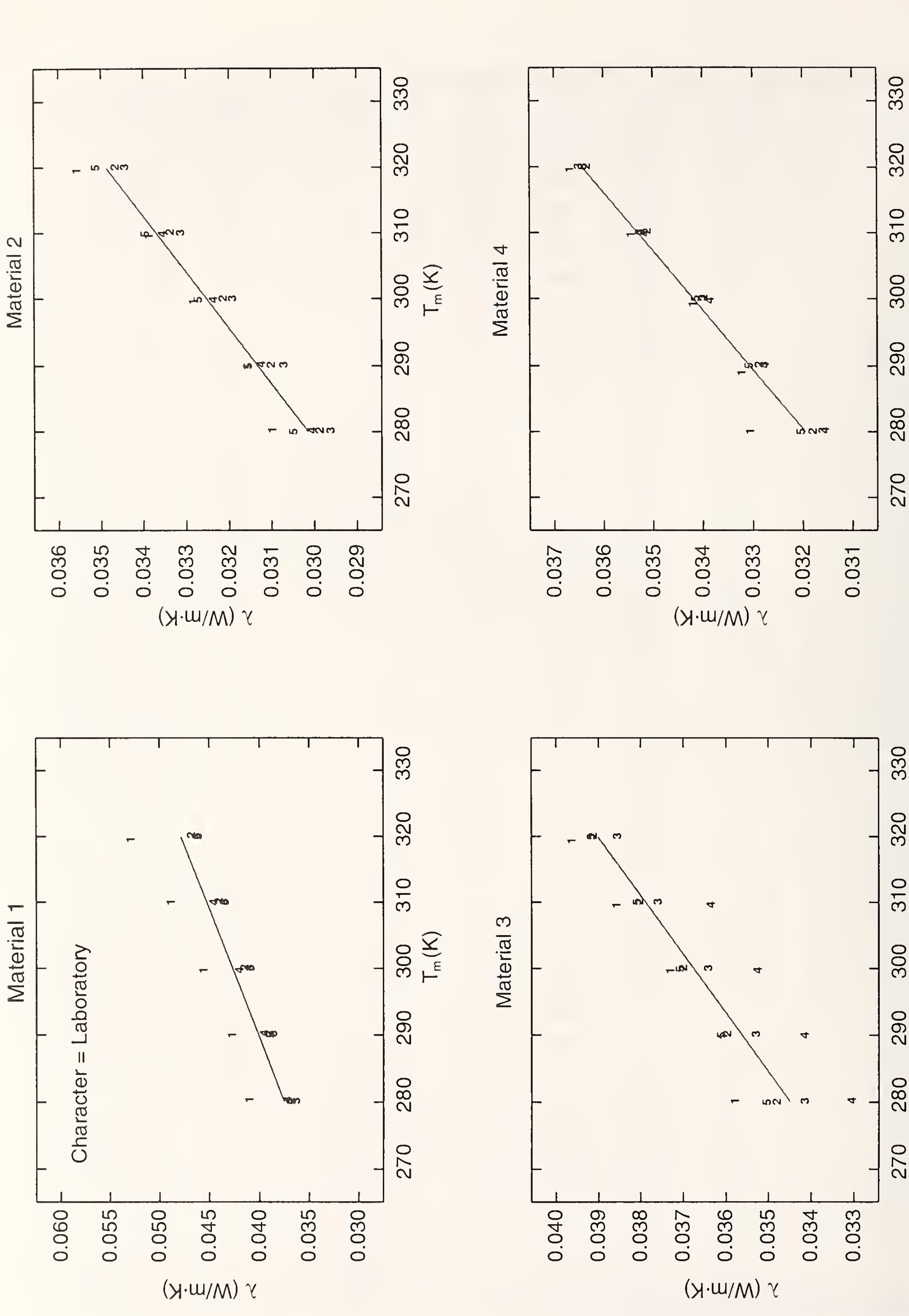


Figure 25. Multi-plot of thermal conductivity as a function of mean temperature (Materials 1, 2, 3, 4)

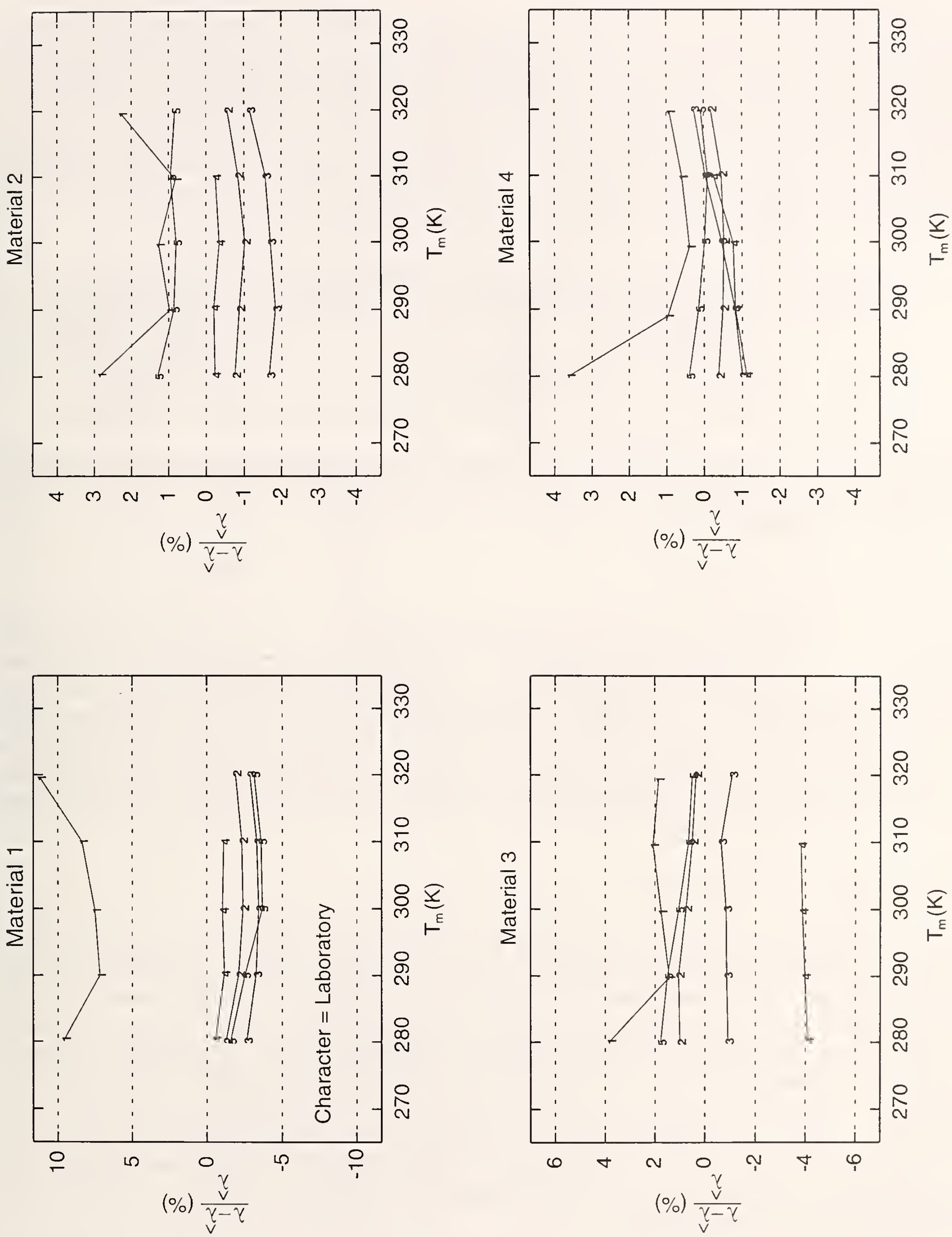


Figure 26. Multi-plot of relative deviations versus mean temperature (Materials 1, 2, 3, 4)

As observed before with the replicate data, the principal conclusion from Figure 26 is that the behavior of the laboratories does, in fact, change from material to material. For the four plots, the location and variation of each set of laboratory data changes from material to material. In short, there is a laboratory-material interaction over the range of temperatures from 280 K to 320 K. By focusing attention again on the extreme values of the data set with respect to location and variation in Figure 26, we will attempt to verify and, possibly, augment the conclusions obtained from the replicate data. Table 24 summarizes the maximum and minimum values, the level of variation (i.e., noise), and apparent outliers for each material.

TABLE 24 –Summary Results of the Interlaboratory Comparison for the Multi-Temperature Data

Material	High Lab	Low Lab	Noisy Lab	Outlying Lab
1	1 (Outlier)	5,3	1	1 (280 K, 320 K)
2	1,5	3	1	1 (280 K, 320 K)
3	1	4 (Outlier)	1	1 (280 K)
4	1	2,3,4	1	1 (280 K)

The results from Table 24 and Table 9 are not identical indicating that there are other interactions between temperature and (some or all of) the factors identified previously. These results are investigated briefly further below.

8.3 Check of Protocol Execution

Figure 27 is a sequence of plots for λ versus T_h, T_c, T_m, ΔT, L, and, q. The first four plots (Figures 27a to 27d) check how effectively the laboratories executed the test protocol (Appendix B) to obtain target temperatures of T_m = 280 K, 290 K, 300 K, 310 K, and 320 K and ΔT = 20 K (regardless of T_m or material). The dashed vertical lines indicate target temperatures. Large horizontal offsets from the dashed lines indicate a laboratory deviation from the test protocol (Appendix B). In general, as seen previously (Figure 15) with the fixed temperature (297,15 K) data, laboratories 1 and 4 appear not to adhere to the test temperature protocol as well as laboratories 2, 3, and 5 (Figure 27d).

8.4 Comparison with Certified Values

Values of $\lambda(T_m, \rho)$ for materials 1 and 4 (SRMs 1451 and 1453, respectively) were determined from Eq 1 using the measured values of T_m and ρ provided by each participant (Table 23). Table 25 summarizes the measurements for materials 1 and 4 for ρ, T_m, ΔT, λ, and computed values of $\lambda(T_m, \rho)$, and the corresponding differences (absolute and relative) between λ and $\lambda(T_m, \rho)$. The data are partitioned by material, laboratory, and run sequence. Note that for material 4, calculations outside the range of certified values 285 K to 310 K (Table 2) are shown as in *italics*. The corresponding uncertainties of ± 3 % and ± 1.3 % for materials 1 and 4, respectively, were used to establish cut-off bounds for the evaluation of anomalous values of λ. The certification equation for material 2 [10] became available after this analysis was completed and is not included. Material 3 is currently undergoing certification process and, therefore, the certification equation is unavailable.

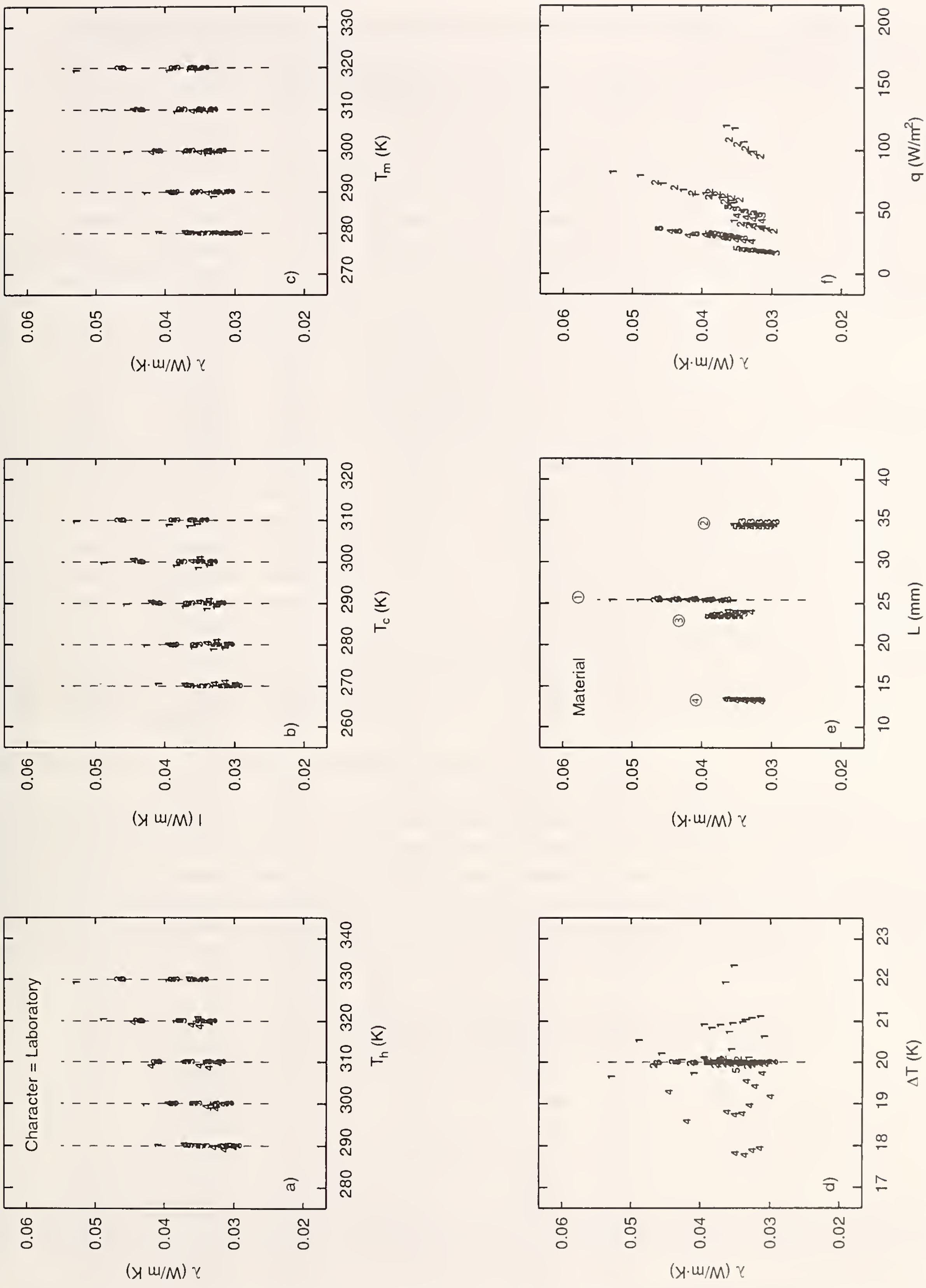


Figure 27. Multi-plot of thermal conductivity versus test parameters (Materials 1, 2, 3, 4)

TABLE 25 – Comparison of Multi-Temperature (280 K to 320 K) Data with Certified Values

Material	Lab	Run Seq.	ρ (kg/m^3)	T_m (K)	ΔT (K)	λ (W/m·K)	λ(T_m,ρ) (W/m·K)	Difference (W/m·K)	Difference (%)
1	1	1	13.35	280.35	19.74	0.04120	0.03757	0.00364	9.68
1	1	2	13.35	289.92	20.05	0.04296	0.03972	0.00324	8.16
1	1	3	13.35	299.79	20.21	0.04583	0.04206	0.00377	8.96
1	1	4	13.35	310.03	20.53	0.04908	0.04463	0.00445	9.97
1	1	5	13.35	319.52	19.66	0.05309	0.04712	0.00596	12.65
1	2	1	14.10	280.12	20.10	0.03707	0.03688	0.00019	0.52
1	2	2	14.10	290.12	20.05	0.03929	0.03904	0.00025	0.63
1	2	3	14.10	300.18	19.97	0.04171	0.04135	0.00036	0.88
1	2	4	14.10	310.18	20.02	0.04428	0.04376	0.00052	1.19
1	2	5	14.10	320.17	19.92	0.04700	0.04630	0.00070	1.52
1	3	1	14.15	280.15	20.01	0.03656	0.03684	-0.00028	-0.77
1	3	2	14.15	290.15	20.00	0.03883	0.03901	-0.00018	-0.45
1	3	3	14.15	300.15	20.00	0.04125	0.04129	-0.00004	-0.10
1	3	4	14.15	310.15	20.00	0.04381	0.04370	0.00011	0.26
1	3	5	14.15	320.15	20.00	0.04654	0.04623	0.00031	0.68
1	4	1	14.13	299.78	18.59	0.0422	0.04122	0.00098	2.37
1	4	2	14.13	310.08	19.28	0.0448	0.04370	0.00110	2.51
1	4	3	14.13	280.39	20.09	0.0374	0.03691	0.00049	1.32
1	4	4	14.13	290.21	20.10	0.0397	0.03904	0.00066	1.69
1	5	1	14.37	280.06	20.01	0.03695	0.03666	0.00029	0.80
1	5	2	14.37	289.97	19.98	0.03908	0.03878	0.00030	0.78
1	5	3	14.37	300.01	19.99	0.04111	0.04104	0.00007	0.16
1	5	4	14.37	310.01	20.00	0.04364	0.04343	0.00021	0.49
1	5	5	14.37	320.02	19.98	0.04637	0.04593	0.00044	0.96
*4**	*1*	*1*	*39.8*	*280.04*	*19.99*	*0.03310*	*0.03158*	*0.00152*	*4.80*
4	1	2	39.8	288.92	20.11	0.03327	0.03262	0.00065	1.99
4	1	3	39.8	299.24	20.98	0.03424	0.03382	0.00041	1.23
4	1	4	39.8	309.72	22.35	0.03549	0.03504	0.00045	1.28
4	*1*	*5*	*39.8*	*319.61*	*21.94*	*0.03673*	*0.03619*	*0.00053*	*1.47*
4	*2*	*1*	*38.6*	*280.11*	*19.98*	*0.03184*	*0.03164*	*0.00020*	*0.62*
4	2	2	38.6	290.14	19.93	0.03292	0.03281	0.00011	0.33
4	2	3	38.6	300.17	19.98	0.03403	0.03398	0.00005	0.15
4	2	4	38.6	310.18	19.94	0.03517	0.03515	0.00002	0.07
4	*2*	*5*	*38.6*	*320.17*	*19.95*	*0.03639*	*0.03631*	*0.00008*	*0.22*
4	*3*	*1*	*38.2*	*280.15*	*20.00*	*0.03164*	*0.03166*	*-0.00002*	*-0.08*
4	3	2	38.2	290.15	20.00	0.03282	0.03283	-0.00001	-0.03
4	3	3	38.2	300.15	20.00	0.03405	0.03399	0.00006	0.17
4	3	4	38.2	310.15	20.00	0.03532	0.03516	0.00016	0.46
4	*3*	*5*	*38.2*	*320.15*	*20.00*	*0.03655*	*0.03632*	*0.00023*	*0.62*
4	4	1	38.8	309.83	17.81	0.0352	0.03509	0.00011	0.30
4	4	2	38.8	299.75	17.77	0.0339	0.03392	-0.00002	-0.06
4	*4*	*3*	*38.8*	*280.13*	*17.94*	*0.0316*	*0.03163*	*-0.00003*	*-0.11*
4	4	4	38.8	290.02	17.88	0.0328	0.03279	0.00001	0.04
4	*5*	*1*	*38.5*	*280.02*	*19.97*	*0.03208*	*0.03164*	*0.00044*	*1.40*
4	5	2	38.5	290.00	19.99	0.03312	0.03280	0.00032	0.98
4	5	3	38.5	300.01	19.96	0.03419	0.03397	0.00022	0.66
4	5	4	38.5	310.01	19.98	0.03528	0.03513	0.00015	0.43
4	*5*	*5*	*38.5*	*320.00*	*19.99*	*0.03647*	*0.03629*	*0.00018*	*0.49*

*Values in *italics* are compared with extrapolated predicted values.

Figures 28a and 28b plot the relative differences of λ and $\lambda(T_m, \rho)$ versus T_m for materials 1 and 4, respectively. The plot character represents the laboratory and horizontal solid lines indicate the cut-off bounds for the (expanded) uncertainty levels for each SRM: ± 3 % for material 1 and ± 1.3 % for material 4. Figure 27a reveals that the relative differences for the multi-temperature data from laboratories 2, 3, 4, and 5 are entirely within the uncertainty levels (± 3 %) for material 1. As was the case for the fixed-temperature replicate data, the multi-temperature data from laboratory 1 was considered sufficiently different (+8.2 % to 12.7 %) from the certification values to warrant rejection as outlying data and, therefore, are excluded from the plot. It is interesting to note that the slopes for most of the laboratories are positive indicating an increasing difference from the certified values at lower temperatures. The change in slope for laboratory 5 at 300 K indicates a temperature interaction. In general, the agreement among these laboratories over the temperature range of 280 K to 320 K is quite encouraging.

Figure 28b reveals that the relative differences for the multi-temperature data from laboratories 2, 3, 4, and 5 are entirely within the uncertainty levels (± 1.3 %) for the certified temperature limit of 285 K to 310 K for material 4 (SRM 1453). For laboratory 5, one data point at 280 K is marginally outside if the uncertainty limits are extrapolated. For laboratory 1, one data point at 290 K is outside the uncertainty limits. The two extreme points for laboratory 1 at 280 K and 320 K are outside if the uncertainty limits are extrapolated. The values at 280 K and 290 K for laboratory 1 are considered sufficiently different from all other data points on the plot to warrant rejection as outlying data. Further examination of the slopes reveals that there is a change in slope (most notably for laboratories 4 and 5) indicating a temperature interaction at lower temperatures. A final conclusion of Figure 28b is that the differences among the laboratories are affected substantially as the mean temperature decreases from room-temperature conditions. Again, the agreement among these laboratories, for most of the data over the temperature range of 280 K to 320 K, is quite encouraging.

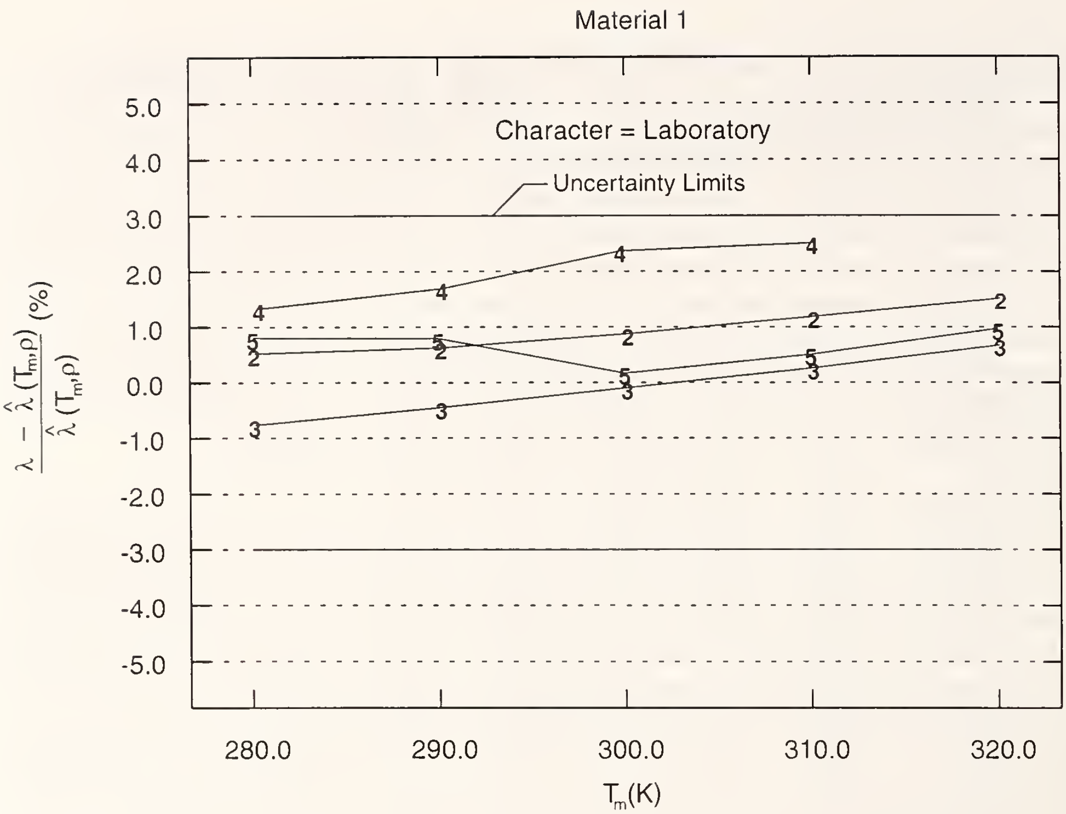
8.5 Comparison with Standard Test Method Precision Indices

At mean temperatures other than room temperature, both ISO 8302 [2] and ASTM C 177 [3] provide statements of uncertainty (precision) statements that range from ± 3 % to ± 5 %. This section evaluates the question: Are the differences among laboratories from 280 K to 320 K significant in comparison with accepted uncertainty and precision statements of the standard test methods for guarded-hot-plate apparatus [2,3]? Further examination of Figure 26, and excluding the outlying data identified in Table 24, yields the ranges summarized in Table 26.

TABLE 26 – Relative Ranges from Figure 26 Excluding Outlying Data Identified in Table 24

	Material 1	Material 2	Material 3	Material 4
Minimum (%)	−3.7	−1.8	−1.1	−1.1
Maximum (%)	−0.6	+1.3	+2.1	+0.9
Range (%)	3.1	3.1	3.2	1.0

The corresponding half-ranges for materials 1, 2, 3, and 4 are ± 1.6 %, ± 1.6 %, ± 1.6 %, and ± 0.5 %, respectively, which are all less than the ISO/ASTM uncertainty (precision) statements of ± 3 % to ± 5 %.



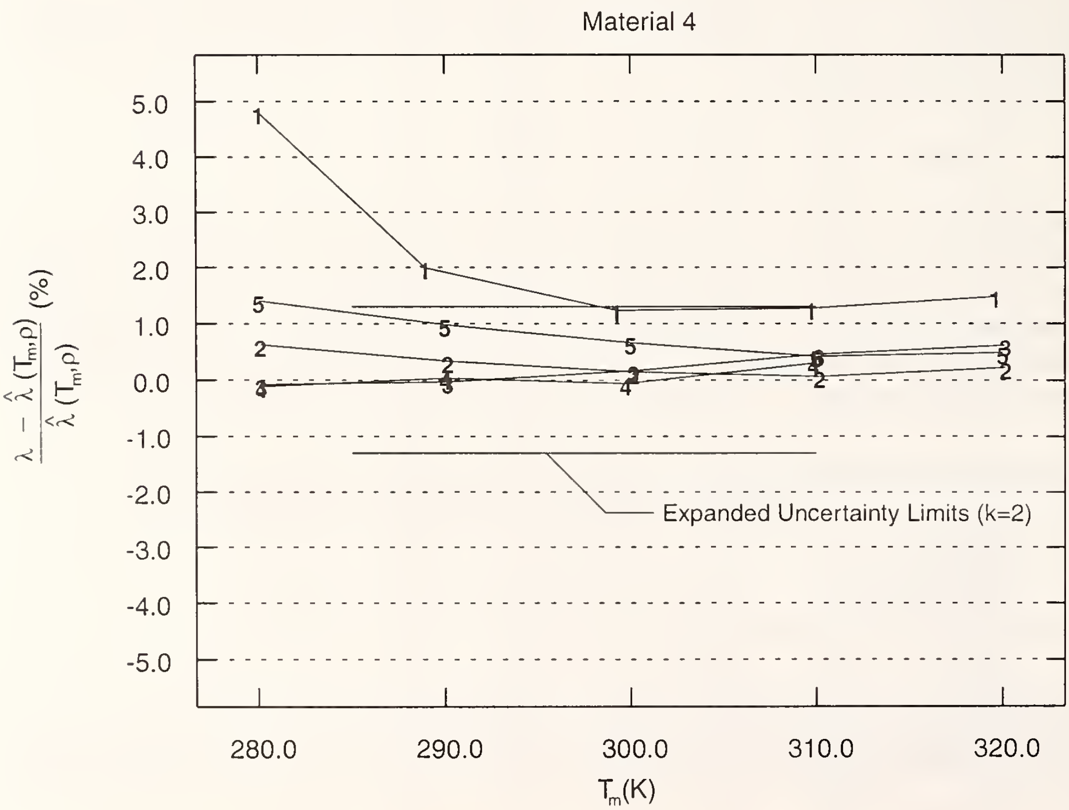


Figure 28. Relative deviations from certified values versus T_m (Materials 1, 4)

9 Conclusions and Recommendations

This international comparison investigated the variability in thermal conductivity results among guarded hot plate laboratories in Canada, France, Japan, United Kingdom, and United States using four national reference materials provided by the participants. The four national reference materials were SRM 1451 (fibrous-glass blanket), IRMM-440 (resin-bonded glass fibre board), JTCCM "candidate" high-density fibrous glass board, and SRM 1453 (expanded polystyrene board). The goal of the comparison was to assess effects of three primary factors – laboratory, material; and, temperature – on the measurement results. The test plan addressed three primary objectives:

1) Characterize the sample of specimens distributed to the participants;
2) Quantify the level of variability of replicate measurements at 297.15 K (24 °C); and,
3) Quantify the effect of temperature over the range from 280 K to 320 K.

The underlying causes for the behavior of the data was investigated using a cause-and-effect diagram that identified 19 (secondary) laboratory factors that may have caused the variation in the test results. The 19 factors were examined individually and collectively using graphical analysis, correlation analysis, and analysis of variance (ANOVA).

9.1 Results

The results from the comparison are summarized as follows.

- The thermal conductivity test data (Figures 4 and 5) indicates that there is a laboratory-to-laboratory difference for each of the materials (except material 4, SRM 1453).

- As expected (and as designed in), there is a material-to-material difference. Material 1, SRM 1451, was the highest thermal conductivity; material 2, IRMM-440, was the lowest. This material-to-material difference was greater than the laboratory-to-laboratory difference.

- The laboratory effect changed from material to material; that is, there is a material-laboratory interaction (Figures 4 and 5).

- The materials differed with respect to variability (Table 12c): material 4 (SRM 1453) was the least variable, followed by material 2 (IRMM-440); material 3 (JTCCM "candidate"); and, material 1 (SRM 1451).

- The dominant factor affecting the laboratory results was procedural in nature. In particular, a significant location and variation shift was experienced by a laboratory that affixed temperature sensors directly to the specimen surface as opposed to permanent sensors affixed to the apparatus plates. This result is in agreement with test results from a previous interlaboratory comparison of guarded hot plate apparatus [15].

- Two fixed temperature (297.15 K) replicate data sets were identified as outlying (laboratory 1, material 1 (SRM 1451) and laboratory 4, material 3 (JTCCM "candidate"). These outliers were respectively identified by 1) comparison with the certified values for SRM 1451; and, 2) laboratory notes indicating a specimen (of material 3) failure (i.e., delamination).

- If the two outlying data sets of the fixed temperature (297.15 K) replicate data are excluded (for reasons noted above), the maximum differences (Table 21) among the laboratories are 1.8 %, 2.7 %, 1.9 %, and 0.69 % for SRM 1451, JTCCM "candidate", IRMM-440; and, SRM 1453, respectively.

- The results of the multi-temperature (280 K to 320 K) data (Table 24) were consistent with the results observed for the fixed-temperature (297.15 K) replicate data. In addition, the results indicated that disagreement among the laboratories tended to increase as mean temperatures decreased from room-temperature (297.15 K) conditions.

- The five laboratories may each be affected differently by the various 19 within-laboratory factors identified herein. The heretofore-presented graphical and statistical analyses are meant to serve as a diagnostic foundation for further perusing by the participants.

9.2 Recommendations

Recommendations are provided for guidance in conducting future interlaboratory comparisons, international or otherwise, as follows.

- Future comparisons of guarded hot plate laboratories should:

  1) Begin planning the interlaboratory comparison with a cause-and-effect diagram. Identify as many of the laboratory factors that differ from laboratory-to-laboratory; and, minimize, by all means possible, the effects of these (secondary) laboratory factors.

  2) In preparing the experimental design, consider the tradeoffs for replicate data at fixed temperature(s) versus single-point data at multiple temperatures. If possible, obtain replicate data at temperatures other than 297.15 K (24 °C).

  3) Specify in the scope the purpose of the comparison, for example, 1) to assess the clarity of a particular test method; 2) to maintain a periodic check of a group of laboratories; 3) to develop precision and bias statements; etc.

  4) Develop an unambiguous test protocol that requires reporting of test data with official test forms. Specify the number of decimal points to be reported for each parameter. Specify whether the bulk density is to be reported for the meter area or entire specimen.

  5) Using the cause-and-effect diagram (Step 1, above) specify what auxiliary information should be reported, for example, conditioning environment, additional information on apparatus in-situ thickness measurement, etc.

  6) Investigate different material types using a balanced number of levels for bulk density for each material type. In anticipation of future comparisons, begin a tracking system to identify new national and regional reference materials.

7) If possible, include additional laboratories (although this will result in increased test time and additional burdens for the coordinating organization).

8) Attempt, if possible, to reduce the cumulative time of the comparison. (Can this 5-year effort (initiation-to-final report) be shortened?)

- The relevant task groups that revise standard test methods for guarded hot plates (ISO 8302 [2] and ASTM C 177 [3]) should:

1) Avoid (or minimize) vagueness in their respective test methods. Standardize the usage of statistical terms in ISO 8302 and ASTM C 177.

2) Develop, as stand-alone documents, standard practices for conducting an interlaboratory comparison with information specific to guarded-hot-plate apparatus. These practices should include standard test protocols, forms for reporting data (including expanded uncertainties (U)), and ancillary information (such as test equipment, etc.). Note that the statistical terminology should be consistent among standard bodies.

3) Specifically, clarify a) the criteria for establishing settling and equilibrium time for taking data so that such information may be standardized; b) the procedure for the measurement of surface temperatures (i.e., when an alternate technique may or may not be utilized); and, c) definition for heat flux.

- In regard to future test materials and matters of equivalence, future international comparisons should be conducted by including either SRM 1453 or IRMM-440.

10. Acknowledgements

The authors gratefully acknowledge the efforts of Dr. Eric S. Lagergren who developed the test plan utilized in this comparison. We also appreciate the cooperation, openness, professionalism, and hard work of the three material characterization laboratories: Japan Testing Center for Construction Materials, National Institute of Standards and Technology, and the National Physical Laboratory; and, the five participating laboratories: Japan Testing Center for Construction Materials, Laboratoire National d'Essais, National Institute of Standards and Technology, National Physical Laboratory, and National Research Council Canada. The authors appreciate the donation of one material (IRMM-440) by the European Commission Institute for Reference Materials and Measurements.

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Appendix A – Participants' Addresses

1) Japan Testing Center for Construction Materials (JTCCM)
Attn: Masayoshi Uezono
Physical Testing Section Central Laboratory
5-21-20, Inari, Soka, Saitama, Japan 340

2) Laboratoire National d'Essais (LNE)
Attn: Gianni Venuti
Departement Energie et Metrologie Thermique
5, Avenue Enrico Fermi
F-78190 Trappes, France

3) National Physical Laboratory (NPL)
Attn: David Salmon
Thermophysical Properties Section
CBTM, Building 16
Queens Road, Teddington, Middlesex, UK TW11 0LW

4) National Institute of Standards and Technology (NIST)
Attn: Robert Zarr
100 Bureau Drive, MS 8632
Building 226, Room B 320
Gaithersburg, MD 20899-8632 USA

5) National Research Council Canada (NRCC)
Attn: Dr. M. K. Kumaran
M-24 chemin Montreal Road
Ottawa, Ontario K1A 0R6 Canada

6) European Commission – Joint Research Centre
Institute for Reference Materials and Measurements (IRMM)
Attn: Dr. Jean Pauwels
Retieseweg, B-2440 GEEL, Belgium

Appendix B – Test Protocols

Protocol for Suppliers of Materials

1) For each material, measure each pair of specimens at a mean temperature of T_m = 24 °C and temperature difference of ΔT = 20 K.
2) Send 1 pair of each material to the participants. Send test results by letter carrier to the address below.

Protocol for Participants

1) Conduct all tests with a single operator.
2) For all 4 materials, one material at a time, conduct 5 independent replicate measurements at a mean temperature of T_m = 24 °C and temperature difference of ΔT = 20 K.
   Note – The replicate measurements are intended to be independent measurements (i.e., capture within-laboratory variability by including typical laboratory/apparatus noise factors). Therefore, the specimen should be removed from the apparatus before proceeding with the next test and conditioned. All pertinent parameters should be fixed as close as possible for each test and the tests should be completed in the shortest time period possible.
3) For all 4 materials, one material at a time, conduct a series of 5 tests at the following mean temperatures: 280 K, 290 K, 300 K, 310 K, 320 K; each test at a temperature difference of ΔT = 20 K.
   Note – These tests are not independent, but should be conducted in a random order.
4) For SRM 1451, compress the low-density, glass fiber blanket insulation (13 kg/m^3) to a test thickness of 25.4 mm using rigid spacer stops to limit plate separation. For the other test materials:
   a) limit the pressure exerted on the specimens by the measuring equipment to 1000 Pa to 2000 Pa; and,
   b) use, when necessary, appropriate spacers to avoid any creep.
5) For each measurement, estimate the relative expanded uncertainty (coverage factor, k = 2), see the ISO *Guide to the Expression Uncertainty in Measurement,* International Organization for Standardization, Geneva, Switzerland, 1993.
   Note – Estimates for relative expanded uncertainty at the same coverage factor (k = 2) are important in order to establish consistent error bars in plotting the data.
6) Complete the appropriate test report for the material tested and when all the measurements have been completed return all test reports by letter carrier to:

   NIST
   Attn: Robert R. Zarr
   Building 226, Room B320
   Gaithersburg, MD 20899 USA

   Note – Test data will be checked initially for consistency (i.e., no missing data, etc.) by R. Zarr and subsequently forwarded to Dr. James J. Filliben for analysis.

Questions?, call or contact Robert R. Zarr, (301)-975-6436, fax: (301)-975-5433, or e-mail: rzarr@nist.gov

Appendix C

MATERIAL CHARACTERIZATION REPORT FORM

INTERNATIONAL INTERLABORATORY COMPARISON

Date: ____________

(To be completed ONLY by supplier of material)

Instructions: The preferred units are indicated. You may use other units, but you must specify the units. Return one form for each material measured. The letters *a* and *b* refer to the pair of specimens tested. Follow the test procedure from either ASTM Test Method C 177 or ISO 8302. Report variations in test procedure, if any.

Laboratory Name: ____

Material: ____

Specimen Characteristics:

	1		2		3		4		5	
Sent to:										
	a	**b**	**a**	**b**	**a**	**b**	**a**	**b**	**a**	**b**
ID #										
m (g)										
x (mm)										
y (mm)										
L (mm)										
ρ ($kg \cdot m^{-3}$)										

Specimen Data (297 K, ΔT = 20 K, 5 pairs):

	1	2	3	4	5
T_h (K)					
T_c (K)					
Q (W)					
L (mm)					
A (m^2)					
λ ($W \cdot m^{-1} \cdot K^{-1}$)					

Comments:

Do you correct specimen thickness for thermal expansion? (circle one) [Y/N]

Do you correct meter area for thermal expansion? (circle one) [Y/N]

Describe plate material, coating or paint, and emittance:

Other (over, or include separate sheet, if necessary):

Appendix D
TEST REPORT FORM
INTERNATIONAL INTERLABORATORY COMPARISON
Date: ____________

Instructions: The preferred units are indicated. You may use other units, but you must specify the units. Return one form for each material measured. The letters *a* and *b* refer to the pair of specimens tested. Follow the test procedure from either ASTM Test Method C 177 or ISO 8302. Report variations in test procedure, if any.

Laboratory Name: ____

Material: ____

Specimen Characteristics:	a	b
Mass (g):		
Size, if circular – average diameter (mm):		
if not circular – average length (mm):		
average width (mm):		
Specimen test thickness, in-situ (mm):		
Specimen bulk density ($kg \cdot m^{-3}$):		

Replicate Data (297 K, ΔT = 20 K, 5 replicates):

	1	2	3	4	5
T_h (K)					
T_c (K)					
Q (W)					
L (mm)					
A (m^2)					
λ ($W \cdot m^{-1} \cdot K^{-1}$)					

Temperature Data (280 K, 290 K, 300 K, 310 K, 320 K, ΔT = 20 K):

	1	2	3	4	5
T_h (K)					
T_c (K)					
Q (W)					
L (mm)					
A (m^2)					
λ ($W \cdot m^{-1} \cdot K^{-1}$)					
T_m (K)					

Comments:

Do you correct specimen thickness for thermal expansion? (circle one) [Y/N]

Do you correct meter area for thermal expansion? (circle one) [Y/N]

Describe plate material, coating or paint, and emittance:

Appendix E – Postscript
Update After Completion Of Analysis Of Data

On August 8, 2001, several of the guarded hot plate laboratory participants met in Cambridge, Massachusetts, USA (in conjunction with the 26^{th} International Thermal Conductivity Conference/14^{th} International Thermal Expansion Symposium) to discuss the analysis of the data in this report, as well as future activities.

With regards to the analysis of the data, a request was prepared for additional information from Laboratory 1, focusing, in particular, on the measurement of specimen thickness and the certification status of material 3. In response, Laboratory 1 indicated that in-situ (or as-tested) measurement of thickness is difficult and that the thickness (l) used for specimen density was the same as the thickness (L) used for the test. In addition, Laboratory 1 indicated that their existing apparatus is old and control is problematic and, therefore, a new guarded-hot-plate apparatus is currently under construction. Information on material 3 indicated that material has now been treated with a surface coating.

With regards to future activities, one of the outcomes of the meeting was the recommendation that future comparisons be conducted as "key comparisons" currently coordinated under the umbrella of the Bureau International des Poids et Mesures (BIPM) as part of the international Mutual Recognition Agreement (MRA). David Salmon from the National Physical Laboratory agreed to pursue the activity. As a direct result of this inquiry, the Consultative Committee for Thermometry (under the BIPM) established a new Working Group 9 on Thermophysical Properties. Further interactions with this working group are anticipated.

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- Structures
- Building Materials
- Building Environment
- Fire Safety Engineering
- Fire Science

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- Advanced Network Technologies
- Computer Security
- Information Access
- Convergent Information Systems
- Information Services and Computing
- Software Diagnostics and Conformance Testing
- Statistical Engineering

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[1]At Boulder, CO 80303.
[2]Some elements at Boulder, CO.

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